QUALITY ASSURANCE PROJECT PLAN AND DREDGE HANDLING PLAN

Lower Menominee River Area of Concern
Menekaunee Harbor Restoration Project
Grant/Project No. GL-00E01312-0 sub 1



Prepared for:

City of Marinette
Wisconsin Department of Natural Resources

October 1, 2014

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Drafted by:

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With final project edits by Donalea Dinsmore, WDNR Office of the Great Lakes

SECTION A – PROJECT MANAGEMENT

A1. Title of Plan and Approval

Quality Assurance Project Plan and Dredge Handling Plan

Lower Menominee River Area of Concern
Menekaunee Harbor Restoration Project

Approvals:

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City of Marinette Project Manager

Date

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Donalea Dinsmore
WDNR Office of Great Lakes Funding and Quality Assurance Coordinator

Date

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Cheryl Bougie
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Date

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Jennifer Conner
EPA-GLNPO Grant Manager

Date

10/2/14

Jared Schmidt, Robert E. Lee & Associates, Inc.
Oversight Consultant Quality Assurance Project Manager

Date

10/2/14

Michael Crystal, Sevenson Environmental Services, Inc.
Construction Project Manager

Date

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Acronyms

AOC	Area of Concern
BOD	Biochemical Oxygen Demand
BMP	Best Management Practice
BUI	Beneficial Use Impairment
GRLI	Great Lakes Restoration Initiative
MDEQ	Michigan Department of Environmental Quality
MDNR	Michigan Department of Natural Resources
MEC	Midpoint Effect Concentration
PAH	Polynuclear Aromatic Hydrocarbon
PEC	Probable Effect Concentration
PHOS	Phosphorus
QAPP	Quality Assurance Project Plan
TEC	Threshold Effect Concentration
TSS	Total Suspended Solids
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
WDNR	Wisconsin Department of Natural Resources
WPDES	Wisconsin Pollutant Discharge Elimination System
WWTP	Wastewater Treatment Plant

A3. Distribution List

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A4. Project/Task Organization

A4.1 Project Organization and Responsibilities

Figure 1 presents the organizational structure for the Menekaunee Harbor Project. All lines of communication, management activities, and technical direction within this project team will follow this organizational arrangement. Any directions or communications from the U.S. EPA will be given to the City of Marinette Project Manager and to the Wisconsin Department of Natural Resources Project Manager. The City of Marinette Project Manager will subsequently communicate directions to Sevenson and Robert E. Lee. The Wisconsin Department of Natural Resources and U.S. EPA project manager will be notified of all proposed changes in personnel. Responsibilities of key project personnel are outlined below and in Table 1.

U.S. EPA Grant Manager

The USEPA Grant Manager (GM) for this project will be Jennifer Conner. The GM has the overall responsibility for general grant oversight to include:

1. Grant administration
2. Final approval of project quality documentation

Wisconsin Department of Natural Resources (WDNR) Great Lakes Funding and Quality Assurance Coordinator

The WDNR Quality Assurance Reviewer for this project will be Donalea Dinsmore.

1. Review and approve the QAPP.
2. Technical consultation on data quality issues, interpretation of laboratory results
3. Communication with USEPA Grant Manager
4. Prepare grant progress reports for USEPA

Wisconsin Department of Natural Resources Project Manager

The project manager for the WDNR will be Cheryl Bougie. The WDNR project manager has overall responsibility for project oversight.

1. Provide technical consultation services to the City of Marinette Project Manager
3. Oversee and address project progress
4. Communicate with the City, WDNR, & USEPA project managers
5. Review progress reports detailing work accomplished
6. Approve project invoices charged to the grant (assistance agreement).
7. Review all final reports

City of Marinette Project Manager

The project manager for the City of Marinette will be Brian Miller. The city project manager has overall responsibility for implementing the project. The city project manager will:

1. Direct project activities.
2. Prepare and submit progress reports detailing work accomplished, funds spent, and the project status.
3. Be responsible for review of project deliverables, development of project planning, and the overview of project strategies.
4. Review site reports for consistency with objectives stated in work plans.
5. Provide final signature on all assessments.
6. Assure timely invoicing to WDNR for eligible project expenses

Construction (Sevenson) Project Manager

The construction manager for this project will be Mike Crystal. The project manager is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The project manager's primary function

is to ensure that technical, financial, and scheduling objectives are achieved. The project manager will:

1. Be responsible for planning, coordinating, monitoring, and evaluating of project field activities.
2. Define project objectives and develop a detailed work plan schedule.
3. Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task.
4. Develop project plans and strategies and review all project deliverables.
5. Review the work performed on each task to ensure its quality, responsiveness, and timeliness.
6. Before sampling, meet with the Team Leader, quality assurance (QA) manager, and field staff to discuss and establish sampling purposes, sampling methodology, number of samples, size of samples, sample preservation methods, chain-of-custody (COC) requirements, analyses required, and which samples will be duplicated in the field.
7. Resolve technical problems.
8. Meet with team members to discuss and review analytical results prior to completion of reports.
9. Responsible for environmental reports and documents.

Robert E. Lee Quality Assurance Manager

The REL quality assurance manager for this project will be Jared Schmidt. The QA manager will:

1. Oversee assessment activities to ensure that sampling methodology, sample preservation methods, and COC procedures are being followed.
2. Assist in any QA issues with field or laboratory questions, as needed.
3. Conduct Field Audits.
4. Maintain a record of samples submitted to the laboratory, the analyses being performed on each sample, the final analytical results, and data validation reports.
5. Prepares Data Assessment Report (DAR).
6. Communicate with the City project Manager, contractor, and WDNR with regard to project progress.

The QA manager's resume is in *Appendix H*

Sevenson Data Manager

The Sevenson data manager for this project will be Scott Burns. The data manager will:

1. Maintain a record of all samples collected and the sample identification information on each sample.
2. Manage data acquired from field assessments and laboratory analyses.
3. Assemble data into computer format.

Sevenson Field Team Leader

The Sevenson field team leader will be Mark Schmitt.

1. Complete on-site Health and Safety Plan (HASP) for each property to be investigated.
2. Be responsible for oversight of field activities and ensure that procedures for the field activities related to the QAPP are executed and documented properly.
3. Submit data generated during field assessment to the data manager.
4. Procuring, coordinating and qualifying all subcontractors.

Sevenson Field Technical Staff

1. Before sampling, meet with Field Team Leader to discuss and establish sampling purposes, sampling methodology, number of samples, sample size, sample preservation

methods, COC requirements, analyses required, and which samples will be duplicated in the field.
2. Be responsible for collection of equipment needed for property assessment work, which would include personal protective equipment (PPE), sampling equipment, sample containers and coolers, water-level meters, monitoring devices, and any other equipment deemed necessary.
3. Implement mandatory health and safety practices.
4. Adherence to work schedules provided by the project manager.
5. Coordination and oversight of technical efforts of subcontractors assisting the field team.
6. Ensure that procedures for the field activities related to the QAPP are executed and documented properly
7. Identification of problems at the field team level, discussion of resolutions with the project manager, and provision of communication between team and upper management
8. Participation in the preparation of the final report

The field technical staff includes Scott Burns Project Engineer/QAQC; Al Lamorticella – General Superintendent; Dan Dragonette – Superintendent; and Paul Jung – CIH/H&S, although other supporting staff from Sevenson may be assigned on an as-needed basis. Resumes for key Sevenson personnel are included in *Appendix H.*

Pace Laboratory Project Manager

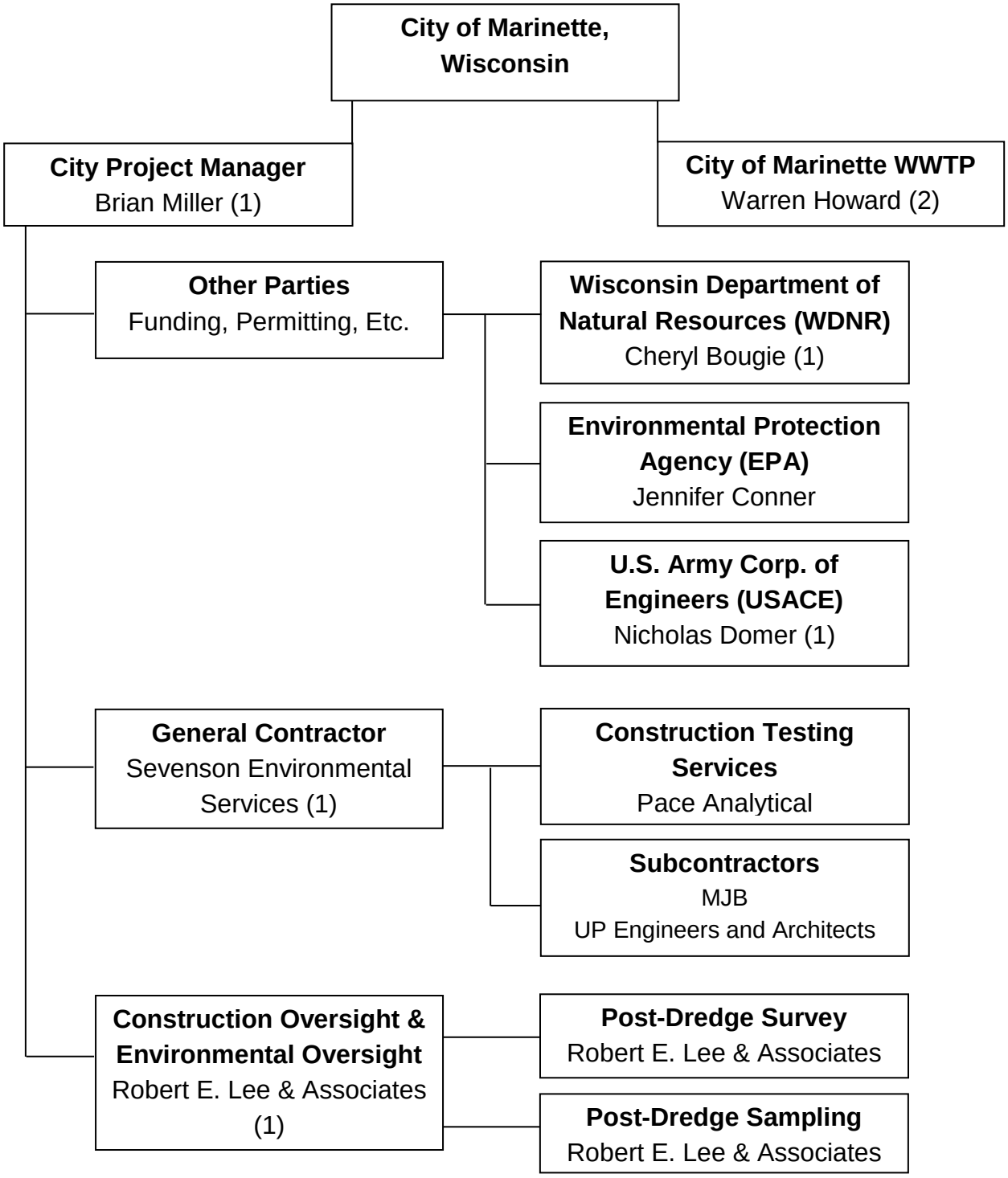
The laboratory project manager for this project will be Dan Milewsky Laboratories.

1. Responsible for samples submitted to Pace Laboratories, including those released to a subcontracted laboratory.
2. Responsible for summarizing quality assurance/quality control (QA/QC) requirements for the project, including those samples analyzed by subcontracted laboratories.
3. Maintain laboratory schedule and ensure that technical requirements are understood by laboratory personnel.
4. Provide technical guidance to Sevenson project manager.
5. Ensure accuracy of the laboratory data.

Table 1. Roles & Responsibilities

Individual(s) Assigned	Responsible for:	Authorized to:
Brian Miller City of Marinette	☐Project management on behalf of City of Marinette	☐Stop work ☐Approve contracts ☐Approve change orders
Cheryl Bougie Wisconsin DNR	☐Project oversight ☐Technical assistance ☐Reporting internally to DNR	☐Stop work
Donalea Dinsmore Wisconsin DNR	☐ Quality Assurance	☐ Approve QAPP
Jennifer Conner US EPA	☐ Grant coordination	☐ Approve funding
Contractor Sevenson Environmental Services	☐Construction activities: dredging, sea wall, containment site ☐Installation and maintenance of stormwater BMPs ☐Installation and maintenance of in-water TSS controls ☐Installation and maintenance of waterway markers ☐Sampling dredge spoils and interstitial water, aggregate, concrete ☐Implementing corrective actions with appropriate documentation	☐Select subcontractors ☐Stop work due to weather ☐Address spills
Construction & Environmental Oversight Robert E. Lee & Associates	☐Performing daily oversight ☐Preparing weekly construction oversight reports ☐Serving as the main point of contact at the site for WDNR/City/engineer	☐Stop work
	☐Performing weekly inspections of stormwater BMPs ☐Performing weekly inspections of in-water TSS controls ☐ Preparing weekly reports, uploading to FTP site ☐ Prescribe corrective actions for BMP's and process ☐Identifying necessary corrective actions and follow up on implementation ☐ Addressing spills ☐Review Compliance sampling, conduct confirmation sampling	☐stop work ☐Prescribe corrective actions for stormwater BMPs

Figure 1. Organization Chart



(1) Authority to stop work.

(2) Authority to recommend stop work

A5. Problem Definition/Background

The Menekaunee Harbor is located within the City of Marinette, Wisconsin, at the mouth of the Menominee River and Lake Michigan's Green Bay (*Figure 2. Project Location*). The harbor lies adjacent to the Michigan-Wisconsin border, and is connected to the Menominee River by a 1,000-foot long navigable channel, though the harbor is currently blocked off at the inlet (located at the Ogden Street Bridge) in order to prevent further contamination from entering the harbor from this "south channel." The harbor is part of the Lower Menominee River Area of Concern (AOC) which includes the lower three miles of the river from the Park Mill Dam to the river's mouth. Beneficial use impairments (BUIs) within the Menominee River AOC include:

1. Degradation of fish and wildlife populations;
2. Loss of fish and wildlife habitat;
3. Restrictions on fish and wildlife consumption;
4. Degradation of benthos;
5. Beach closings; and
6. Restrictions on dredging activities.

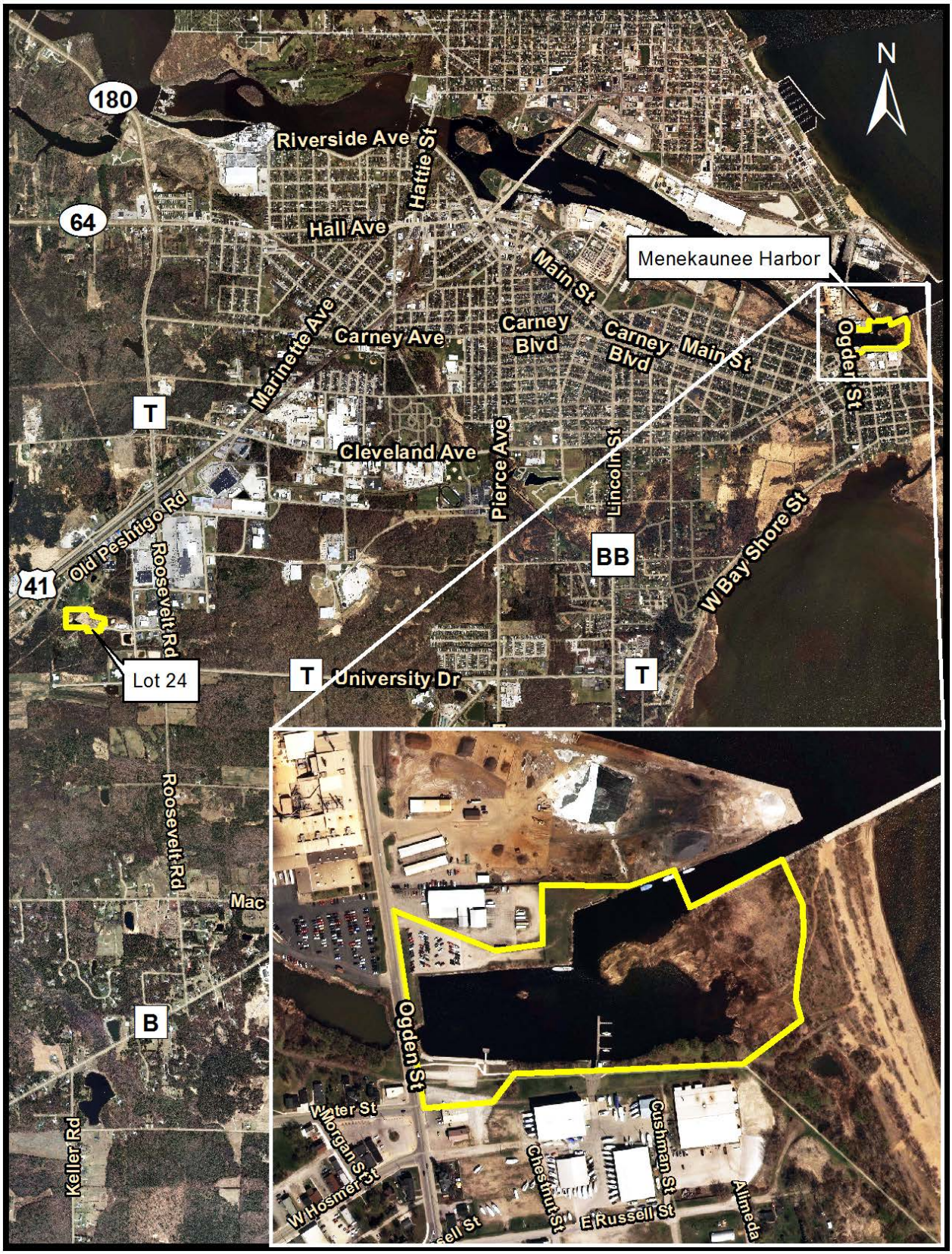
Historically the Menekaunee Harbor extended directly eastward to the shoreline of the Green Bay and was an extension of the Lower Menominee River. However, sand dunes formed on the east side of the harbor following construction of the government pier, establishing a natural barrier that protects the area from lake and storm activity. These geologic and hydrologic conditions support a formerly-diverse wetland complex that extends from the east pocket of the Menekaunee Harbor eastward toward the Green Bay. The shallow waters, submerged vegetation, and wetlands provided diverse and critical habitat for a variety of fish, birds, and other wildlife.

However, hydrologic alteration has caused extensive sediment deposition which not only negatively impacted the diversity and function of the wetland complex, but has also severely restricted navigation within the harbor. Additionally, pollutants associated with historical industrial manufacturing practices along the river have resulted in degradation and contamination the harbor, and contribute daily to the cause of the BUIs within the Menominee River AOC. In addition to contaminants within the sediment of the harbor and shallow depths caused by sediment, the existing timber pile shoreline (wooden seawall) treatment is in poor condition and is in need of replacement.

The City of Marinette, working in tandem with the Wisconsin Department of Natural Resources and the U.S. Environmental Protection Agency (U.S. EPA) has received funding support to address the dredging, seawall and habitat restoration activities associated with the Harbor and Menominee River Area of Concern.

The purpose of the project is to address BUIs within the harbor by removing contaminants, restoring the fish and wildlife habitat and at the same time improving navigation and shoreline treatments. This QAPP addresses dredging (both contaminant- and navigation-related) and sea wall replacement.

Figure 2. Project Location



A6. Project/Task Description

The Menekaunee Harbor Improvement project will dredge the harbor to a navigable depth of 8 feet during historic low lake levels—568.0 mean sea level (MSL)—in all locations to remove contaminants at or above the Threshold Effect Concentration (TEC) for arsenic, copper, lead, mercury, and/or zinc (*see Appendix A – Plan Set and Appendix B – Sevenson Work & Dewatering Plan*). Deeper dredging depths will be achieved in areas where contaminants have been located below 568.0 msl. The non-navigational areas on the eastern side of the harbor will be backfilled to 576.0 msl to provide final water depths of approximately two feet for habitat restoration purposes. Clean dredge spoils (termed "beneficial-use fill") removed from the west side of the harbor will be used to bring the east side to the desired restoration depths. An estimated 27,500 cubic yards of Environmental and 47,500 cubic yards of Navigational Dredging (22,500 cubic yards which will be beneficial re-use within the harbor) are anticipated. Elevations and referenced in this document are based on survey data of the Menekaunee Harbor with control from the Corps of Engineers benchmark located near Ogden Street as indicated on the drawings. The Wisconsin County Coordinate System (WCCS), Marinette Zone, North American Datum of 1983 – 1991 Adjustment (NAD83(91)) was used for horizontal control, and the North American Vertical Datum of 1988 (NAVD88) for vertical control.

The contractor plans to complete the project by use of mechanical and hydraulic dredging. Hydraulic dredging will be used to complete the beneficial-use portion of the dredging and mechanical dredging will be used for all material disposed of off-site. The contractor will allow the material to dewater naturally by placing, working, and blending the material on a dewatering pad constructed in the staging area. The contractor will have Portland cement and appropriate equipment on site to blend with the materials if necessary for dewatering. All wastewater will be collected and treated by an on-site facility and disposed into the sanitary sewer lines to be delivered to the City of Marinette WWTP. Woody debris encountered during navigational dredging will be shredded at Lot 24. All other large trash and debris items will be transported to the landfill.

The existing shoreline timber pile wall removal and replacement will take place in conjunction with the dredging operations as dredging along the existing timber pile walls may cause the walls to become undermined and the shoreline to collapse. The existing timber pile walls are to be cut off at a minimum of two feet below final grade and covered with beneficial-use fill and/or riprap as indicated on the plans. The project includes shoreline improvements consisting of approximately 1,100 feet of riprap installations, 200 feet of anchored sheet pile wall, and 150 feet of stepped ledge stone. The remaining shoreline (to the east side of the harbor) will remain in its natural state, as there is currently no timber pile or riprap. The concrete fishing steps located in the Southeast corner of the harbor will remain in place.

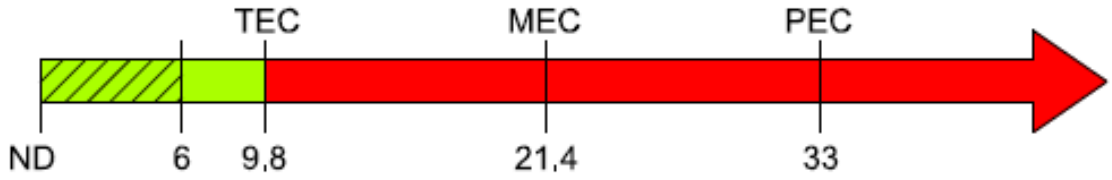
The project also involves development of a dredge spoils containment site at Lot 24, located at the west end of Murray Street on the west side of Marinette. This containment site footprint is approximately three acres, and has the capacity to hold up to 54,500 cubic yards of dredge spoils. Only navigational dredge spoils with an arsenic concentration less than 6.0 ppm will be accepted at this site. There is an expected volume of 22,500 cubic yards of material meeting this restriction. Figure 3 (Dredge Spoils Management Criteria) shows how different classes of spoils will be handled. Material sent to Lot 24 is required to contain less than 6 ppm (less than the 9.8 ppm TEC limit) per the DNR Exemption for Low Hazard Waste. Material between 6 ppm and 9.8 ppm may still be used for ecological restoration as "beneficial-use fill".

A tentative sequence of events is provided in the table on the following page. The final construction schedule will ultimately be determined by the selected contractor, but any sequence disruptions should be approved by the project manager.

Table 2. Tentative Project Schedule

Task	Start Date	End Date	Notes
Bid Period	06/09/2014	07/18/2014	
Pre-bid Meeting	06/24/2014	06/24/2014	
Bid Addenda Issued	07/11/2014	07/11/2014	
Bid Opening Date	07/18/2014	07/18/2014	
Project Management Team Review	07/18/2014	08/04/2014	
Contract Awarded	08/05/2014	08/05/2014	
Contracting, Notice to Proceed	08/08/2014	08/08/2014	
In-Stream Work Restriction Ends	06/15/2014	06/15/2014	Cannot work in water until after June 15 – Post Fish Spawning Window
Mobilization	08/18/2014	08/22/2014	Arrange staging and dewatering areas, fencing
Erosion Control Installation	08/18/2014	08/22/2014	Must occur prior to land disturbance – see SWPPP
Clearing and Grubbing	08/22/2014	11/01/2015	Preparation of Lot 24 containment site
Dredging	08/22/2014	11/01/2015	Includes dewatering and hauling, potential for drying agent mixing, containment of temporary storage piles for curing and segregation of sediment
Shoreline Treatment	08/22/2014	11/01/2015	
Backfill East Portion of Harbor	10/01/2014	11/01/2015	
Permanent Stabilization	10/01/2014	11/01/2015	
Equipment Decontamination	10/01/2014	11/01/2015	
Demobilization	11/01/2014	11/01/2015	

Figure 3. Dredge Spoils Management Criteria



DISPOSAL BASED ON ARSENIC LEVEL

ALL UNITS IN mg/Kg/(ppm)

USING TOTAL METALS CONCENTRATIONS

	TEC	MEC	PEC
ARSENIC	9,8	21,4	33
COPPER	32	91	150
LEAD	36	83	130
MERCURY	0.18	0.64	1.1
ZINC	120	290	460

*<6,0 Arsenic Proposed for Lot 24 acceptance

LEGEND

= REMAIN IN HARBOR FOR ECOLOGICAL RESTORATION

= TO LOT 24 CONTAINMENT SITE

= TO LANDFILL

ND = NOT DETECTED

A7. Quality Objectives & Criteria

A7.1 Data Quality Objectives (DQOs)

DQOs are qualitative and quantitative statements that clearly state the objective of a proposed project, define the most appropriate type of data to collect, determine the appropriate conditions for data collection, and specify acceptable decision error limits that establish the quantity and quality of data needed for decision making. The DQOs are based on the use of the data that will be generated. Different data uses may require different quantities of data and levels of quality.

The overall project objectives include:

- Creating aquatic habitat area on the eastern side of the harbor with final water depths of approximately two feet (sediment elevation of 576.0 msl with clean sediment)
- Removing contaminated sediment from the harbor such that target metals concentrations in surface sediments (top 6 inches) do not exceed their toxic effects concentrations (TECs) as indicated in figure 3.
- Properly dispose of sediments with concentrations above the TEC
- Properly dispose of interstitial water (wastewater) from removed sediments
- Handle other navigational dredge spoils appropriately and cost-effectively per Low Hazard Waste Exemption
- In navigational areas of the harbor, achieve an elevation no higher than 568.0 msl (per technical specifications) so a navigational depth of 8 feet exists at historic low lake levels with stable side slopes.

Carriage/Interstitial Water – Wastewater Treatment
Wastewater generated from dewatering will be eliminated into the sanitary sewer system of the City of Marinette Wastewater Treatment Plant. The WWTP requires that this water shall have contaminant levels at or below the plant's local limits (*Table 3*). Contaminant levels shall be verified by grab samples collected and tested as described in Section B. This sampling and analysis falls within the pretreatment requirements of the Wisconsin Permit Discharge Elimination System (WPES) and data quality must meet requirements specified in NR 219 Wis. Admin. Code. In the event that approval for direct discharge of wastewater is desired, the contractor must apply for a WPDES permit and comply with the associated discharge limits.

Table 3. Wastewater Treatment Plant Limits

METALS		
Arsenic	mg/l	0.10
Cadmium	mg/l	0.14
Chromium	mg/l	2.41
Copper	mg/l	1.40
Lead	mg/l	2.02
Mercury	mg/l	0.01
Nickel	mg/l	1.35
Selenium	mg/l	0.12
Silver	mg/l	0.50
Zinc	mg/l	2.25
GRAB SAMPLES		
Cyanide	mg/l	0.19

Oil & Grease	mg/l	100
Phenols	mg/l	25
pH		6.0-9.0
SURCHARGEABLES		
BOD	mg/l	300
TSS	mg/l	350
PHOS	mg/l	14.5

Contaminated Sediment Removal
Sediments contaminated with metals above the TEC (Figure 3) are to be removed so surface sediments (top 6 inches) remaining in the harbor are below the associated TEC value as demonstrated by sampling and analyses

In general, spoils with more than 30% fines (particles smaller than the #200 sieve) contain contaminants above the TEC and are designated Environmental Spoils *for removal and landfill disposal.*
.
Remaining spoils (generally with less than 30% fines) are anticipated to be Navigational Spoils that, based on contaminant concentrations, may either be beneficially used in the ecological restoration area or removed for placement in Lot 24 . To be acceptable for beneficial reuse in the ecological restoration area, sediments must have target analyte concentrations below the following:

9.8 mg/kg arsenic;
32 mg/kg copper;
36 mg/kg lead;
0.18 mg/kg mercury
120 mg/kg for zinc.

Navigational spoils for upland placement at Lot 24 must have concentrations below 6 mg/kg arsenic as determined post removal (i.e. stockpile samples).

To balance staging considerations for dredging, costs, and schedule; sediments targeted for beneficial use may be sampled in situ and demonstrated to meet sediment quality in the top 24 inches of targeted sediment either prior to dredging or following dredging of environmental spoils (overburden).

Sediment for Landfill Disposal
The landfill has accepted the existing chemical characterization and material for disposal must meet landfill requirements for physical characteristics described in Appendix E. Testing includes percent moisture, grain size, liquid limit, plastic limit, plasticity index, and hydraulic conductivity. The landfill may waive testing requirements at their discretion.

Navigation
The final post-dredge contours will result in a navigable depth of at least 8 feet (568.0 msl) during current historic low lake levels. This excludes the ecological restoration area on the east side of the harbor, where the final depth should be around 2 feet (574.0 msl). An initial survey will be conducted by REL (on NAVD88 DATUM) to develop a baseline topographic survey. Bi-weekly checks will be also be conducted by REL to validate progress payments of material removed by dredging. Progress surfaces will be created to determine the volume of material removed during each period between surveys. Additionally dredge volumes will be

cross-checked with communication of tonnage disposed of at the landfill, and initial and progress surveys and volume checks at Lot 24. Surveys will be performed before and after bulk removal, then again after final placement of beneficial reuse material in the restoration area. A post-dredge hydrographic survey shall be completed to verify tolerance and confirm pay quantities. Bathymetric surveys will be completed as soon as possible after final dredge depths and/or final grades are achieved. This data shall be accurate to within ±24 inches horizontally, and ±3 inches vertically.

Surface Water
Operational controls (Best Management Practices) must be in place to control storm water per the NR 216 construction site storm water discharge permit and the NR 151 Technical Standards as found in Appendix I (Harbor) and Appendix J (Lot 24). Assessments for meeting the permit conditions will be based on proper installation and maintenance based on the weekly and qualifying rain event inspections. Water controls prevent visible discharges of suspended solids beyond the work area in accordance with the Chapter 30 permit located in Appendix K. Assessments for meeting permit conditions will be based on visual observation.

Shoreline Treatment
Shoreline treatments shall be repaired or replaced incidental to dredging activities. Sevenson has two subcontractors (MJB & UP Engineers & Architects) who will be addressing the technical requirements for concrete and shoreline installation and testing. UPEA will be handling concrete bearing capacity testing and Proctor and compaction testing for shoreline stability. Refer to *Appendix C* for technical specifications and Sevenson certifications for subcontractors with regard to concrete and Proctor testing protocol.

A. Riprap Shoreline Treatment
Riprap and riprap bedding to meet gradation/size requirements outlined in specifications.

B. Sheet Pile Wall Shoreline Treatment
- Concrete for deadman wall will be tested for strength. Loose soil (N<5 BPF) within 3 feet of the bearing elevation and 10 feet of the wall side of the concrete deadman will be removed and re-compacted.
- Soils having more than 5% organics will be removed from within 5 feet of the bearing elevation. Sevenson will be removing topsoil to accommodate shoreline treatments. Previous geotechnical reports indicate soils with high sand content and minimal organics, restricting the requirement for independent sampling of soil physical qualities during construction activities.
- Concrete testing will be performed in accordance with specifications section 03 30 00.
- Anchor rod tensioning will be testing in accordance with specifications section 05 50 00.

C. Ledge Stone Shoreline Treatment
- Ledge stone shoreline treatment shall meet sizes and tolerances outlined in specifications section 31 37 12 and shown on the Drawings.

D. Unfinished Shoreline Treatment
- Shoreline with no treatment shall meet grading tolerance outlined in specifications section 31 22 00.

A7.1.1 Analytical Quality Objectives

Analytical quality objectives are used to ensure that the analysis will accurately and adequately identify the contaminants of concern, and to ensure that the analysis selected will be able to

achieve the quantitation limits less than or equal to the target cleanup levels.

A7.2 Quality Assurance Objectives for Measurement

The overall QA objective for this portion of the construction project is to develop and implement procedures for field sampling, COC, laboratory analysis, and reporting using WDNR protocol. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventative maintenance of field equipment, and corrective action are described in other sections of this QAPP.

Data quality objectives for measurements during this project will be addressed in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). The numerical PARCCS parameters will be determined from the project DQOs to ensure that they are met. The DQOs and resulting PARCCS parameters will require that the sampling be performed using standard methods with properly operated and calibrated equipment, and conducted by trained personnel.

A7.2.1 Precision

Precision is the degree of agreement among repeated measurements of the same parameter under the same or similar conditions. Precision is reported as either relative percent difference (RPD) or relative standard deviation (RSD), depending on the end use of the data.

A7.2.1.1 Field Precision Objectives

Field precision will be assessed through the collection and analysis of field duplicate samples. RPDs will be calculated for the detected analytes from investigative and field duplicate samples. Water matrix samples can be readily duplicated due to their homogeneous nature; sediment deposits can be more heterogeneous even for metals. Due to this difficulty, RPDs of ±25 percent for wastewater grab samples and ± 40 percent for metals in sediments will be used as advisory limits for analytes detected in both investigative and field duplicate samples at concentrations greater than or equal to five times its quantitation limit. A summary of duplicate samples to be collected is presented in *Table 6.* Per the WDNR, field duplicate samples must be provided for each matrix sampled. The minimum number of field duplicate samples required for each round of sampling is one for every 20 samples. If there are fewer than 20 samples per matrix, one field duplicate per matrix will be submitted.

A7.2.1.2 Laboratory Precision Objectives

For Pace Laboratories, precision of laboratory analyses will be based upon laboratory matrix spike/matrix spike duplicate (MS/MSD) analyses. Precision is reported as RPD or RSD, and the equation to be used to determine precision is below. MS/MSD analyses will be either at a rate of 1 per 20 samples/matrix received by the laboratory or in accordance with laboratory Standard Operating Procedures (SOPs). In Appendix G lists the MSD and RPDs used by Pace Laboratories.

$$\text{RPD} = \frac{\left| R_1 - R_2 \right|}{(R_1 + R_2)/2}$$

X 100

where: R_1 = value of first result; R_2 = value of second result.

A7.2.2 Accuracy

Accuracy is the extent of agreement between an observed or measured value and the accepted reference, or true, value of the parameter being measured.

A7.2.2.1 Field Accuracy Objectives

The objective for accuracy of the field sample collection procedures will be to ensure that samples are not affected by sources external to the sample, such as sample contamination by ambient conditions or inadequate equipment decontamination procedures. Sampling accuracy will be assessed by evaluating the results of equipment blank samples for contamination.

Equipment blanks will be collected by pouring laboratory-prepared water or distilled water over or through the field sampling equipment and collecting the rinsate in the proper analytical containers. Equipment blanks must be submitted to the laboratory with investigative samples and analyzed for the same parameters as the investigative samples. The minimum number is 1 per 20 field samples per matrix or, if less than 20 samples are collected, one equipment blank per day per sample matrix. If all disposable or single use sampling equipment is used, then a field blank will be collected in lieu of an equipment blank at a rate of one per sampling event or per lot of bottles, whichever is more frequent.

Equipment and /or field blanks will be analyzed during assessment activities in order to assess potential problems as they occur.

A7.2.2.2 Laboratory Accuracy Objectives

Laboratory accuracy will be assessed by determining percent recoveries from the analysis of laboratory control samples (LCSs). The analyses of MS/MSD samples are also utilized to determine laboratory accuracy by determining percent recoveries from the analysis of MS/MSD samples. MS/MSD samples will be collected for organic and inorganic analyses at a minimum frequency of 1 per 20 or fewer samples. The accuracy of the organics analyses also will be monitored through analysis of surrogate compounds. Surrogate compounds are added to each sample, standard, blank, and QC sample prior to sample preparation and analysis. Surrogate compounds are not expected to be found occurring naturally in the samples, but behave analytically similar to the compounds of interest. Consequently, surrogate compound percent recoveries will provide information on the effect that the sample matrix exhibits on the accuracy of the analyses.

In addition, please see Section 5.0 of Pace's Laboratory's QA Manual, *located in Appendix C* of this QAPP, for the laboratory's QA objectives.

A7.3.1 Representativeness

Representativeness is a qualitative term that describes the extent to which a sampling design adequately reflects the environmental conditions of the site. It also reflects the ability of the sample team to collect samples and laboratory personnel to analyze those samples in such manners that the data generated accurately and precisely reflect the conditions at the site. Permit conditions and beneficial reuse approvals specify the frequency for sampling of waste

materials.

A7.3.1.1 Measures to Ensure Representativeness of Field Data

Representativeness will be achieved by establishing the level of allowable uncertainty in the data and then statistically determining the number of samples needed to characterize the population through the DQO process. For confirmation sampling in areas where sediment will remain in the harbor, surface samples will be collected once target elevations are reached at a rate of one sample per 2500 square feet. The oversight consultant will establish a sampling grid in the area and then select random locations so samples are representative of the dredge area. In the habitat restoration area, confirmation samples may be composited to assure that the arsenic concentrations are representative of the full surface area. Surface samples are intended to represent the benthic layer so the preferred sampling methods will recover the top six inches. If the Ponar sampling is employed for post-dredge sample collection, the capture in the dredge must be evaluated to determine whether the sample represents the six inch benthic zone rather than skimming the surface

A7.4.1 Completeness

Completeness is defined as the measure of the quantity of valid data obtained from a measurement system compared to the quantity that was expected under normal conditions. While a completeness goal of 100 percent is desirable, an overall completeness goal of 90 percent may be realistically achieved under normal field sampling and laboratory analysis conditions.

A7.5.1 Comparability

The confidence with which one data set can be compared to another is a measure of comparability. The ability to compare data sets is particularly critical when a set of data for a specific parameter is compared to historical data for determining trends. The data generated for this project is designed to be compared to general sediment quality goals and wastewater permit compliance. Elevation measurements must be comparable to navigational standards and any discrepancies in the initial bathymetry between the Contractor and Construction and Environmental Oversight firm must be resolved to assure project performance and appropriate payment.

A7.6.1 Sensitivity

Sensitivity is the ability of a method or instrument to detect a parameter to be measured at a level of interest.

A8. Special Training/Certification

Construction personnel shall be required to partake in a training session on best management practices (BMPs) associated with the project SWPPP. This training will be conducted immediately following mobilization. Topics will include: installation and maintenance of stormwater controls; procedures for spill prevention and response; procedures for storage, handling, and disposal of construction products, materials, and wastes. Additionally tailgate training sessions will be held regularly by the construction contractor to ensure that construction personnel remain mindful of environmental, safety, and quality considerations.

Contractor shall have HAZWOPER/OSHA 40 hour training certification. Sevenson personnel and subs who will be working with hazardous materials will have the required 40 hour training (29CFR1910.120) and 8 hour refresher. Sevenson employees and subs who will be performing the sample collection, labeling, preservation, packaging and shipping of samples will be trained and sign off on the SOPs and HASP requirements. We anticipate water and sediment sampling will be performed by Scott Burns, he holds 40 Hr OSHA training + 8hr refresher as well as Army Corps QC training. Scott will be trained and sign off on the pertinent sampling/handling SOPs prior to the sampling events. Should another sampler be used that person will also have 40 Hr training and be trained on the SOPs prior to performing the work.

Construction oversight personnel must be licensed in Wisconsin as a Professional Engineer (PE), have designation as an Engineer in Training (EIT), or be a Senior Engineering Technician with direct oversight by a PE, and have experience or training in construction observation, dredging, concrete reinforcement inspection, sheet pile wall installation.

Environmental compliance personnel shall have had training in the principles and practices of erosion and sediment control measures, and possess the skill to assess conditions that could impact storm water quality and to assess the effectiveness of any sediment and erosion control measures that are in use. Licensure as a Professional Engineer or registration as a Certified Professional in Erosion and Sediment Control (CPESC) or similar is preferred.

Robert E. Lee & Associates, Inc. (REL) personnel conducting bathymetric, topographic surveys and quality assurance sampling have the required knowledge of SOPs and site specific HASP requirements. REL employees who will be performing the QAQC sample collection have completed the required 40-hour training (C29CFR1910.120), and will sign off on the SOPs and HASP requirements.

Any laboratories analyzing samples of dredged sediments and/or interstitial water must have current WDNR laboratory certification for all of the contaminants associated with the harbor and will be certified as required by applicable state and/or federal agencies for the fields of testing relevant to the requirements of each individual project. The laboratory selected for the majority of the analytical work required for this project is Pace Laboratories located in Green Bay, Wisconsin. Pace Laboratories has been certified under the Wisconsin Certification Program NR 149 Wisconsin Administrative Code which is administered by the State of Wisconsin. Pace Laboratory's Certification number is 405132750 – Green Bay (oil and grease Pace Laboratory 999407970 - Minnesota). Dan Milewsky will serve as the Pace Laboratory project manager and QA manager and will be ultimately responsible for ensuring the quality of the laboratory data.

Personnel performing post-construction topographic surveys shall have Wisconsin land surveyor licensure.

It is preferable that personnel performing the post-dredge bathymetric survey shall be licensed in Wisconsin as either a land surveyor or a PE, and has familiarity with collection of spatial data.

A9. Documents and Records

A private FTP site maintained by Robert E. Lee & Associates, Inc. shall be made accessible to all parties involved in the project and shall be updated as necessary with the most current approved versions of the project plan drawings, QAPP, and Erosion Control & Storm Water Management Plan. This site will also be utilized for dissemination of progress reports, audit reports, construction observation logs, and stormwater inspection records. An email notification shall be sent to personnel identified in A3. Distribution List upon upload of any updated plans. This FTP site will also provide templates for forms used by field personnel. In addition to the

documents outlined below, additional submittals and documentations as required by the technical specifications shall also be prepared and distributed.

Documentation will be prepared throughout the project for the following:

1. Health and safety program implementation
2. Schedule, budget, and personnel details
3. Conformance to project specifications
4. Stormwater management practices implementation
5. Dust control management practices implementation
6. In-water TSS Controls/BMPs
7. Surveying of containment site topography
8. Construction quantities management
9. Materials and equipment taken off site and brought on site
10. Photographs and written records management
11. Construction materials utilized, including those for erosion and sediment control, and seed mixes placed
12. Site challenges and actions taken to address.

The documentation gathered during site construction, operations, and closure, including as-constructed drawings, will be assembled into a project documentation report that will be provided to the WDNR following cessation of harbor site and containment site activities and closure. Three hard copies and one electronic CD copy will be provided to both the WDNR and the City of Marinette.

Construction observation logs shall include details and photographs for daily construction activities. These logs shall be retained for the duration of the project plus at least three years post-construction. Construction observation shall be used to produce reports which include documentation of all of the following:

1. Sediment sample results
2. Wastewater sample results
3. Stormwater inspection reports
4. In-water TSS controls reports
5. Construction as-built drawings
6. Confirmation hydro survey maps
7. Material quantities for each location

Stormwater inspection records shall include details on installation, maintenance, and deficiencies of stormwater BMPs, as well as photographs. These inspection records must be retained for the duration of the WPDES general permit coverage plus at least three years from the data of notice of termination, per NR 216.48. Stormwater inspection records shall include all of the following:

1. The date, time and location of the construction site inspection.
2. The name of the individual who performed the inspection.
3. An assessment of the condition of erosion and sediment controls.
4. A description of any erosion and sediment control best management practice implementation and maintenance performed.
5. A description of the present phase of land disturbing construction activity at the construction site.

SECTION B – DATA GENERATION & ACQUISITION

B1. Sampling Process Design (Experimental Design)

B1.1 Sampling Process Design

Interstitial Water – Wastewater to Sanitary Sewer
Marinette Wastewater Treatment Plant (WWTP) has agreed to accept wastewater from these dredging operations. In accordance with the agreement with the WWTP, influent and effluent samples will will be collected. The influent samples will be taken at the entrance to the first Influent Equalization Tank. Effluent samples will be taken at the Bag Filter Effluent Sample Port. into tanks and the wastewater. Grab samples will be collected at the frequency outlined in *Table 3 and in Appendix D – Wastewater Treatment System Monitoring Plan*. Sevenson will be submitting the sampling results directly to the WWTP manager and keeping the A/E (REL) informed throughout the sampling process. There has been no operator certification requirement requested by the WWTP manager with regard to sampling requirements. Sevenson's staff (Scott Burns) will be conducting sampling activities during the project period; his credentials are listed in the resumes under *Appendix H*

Sediment

Four types of sediment sampling are planned to accomplish project objectives:

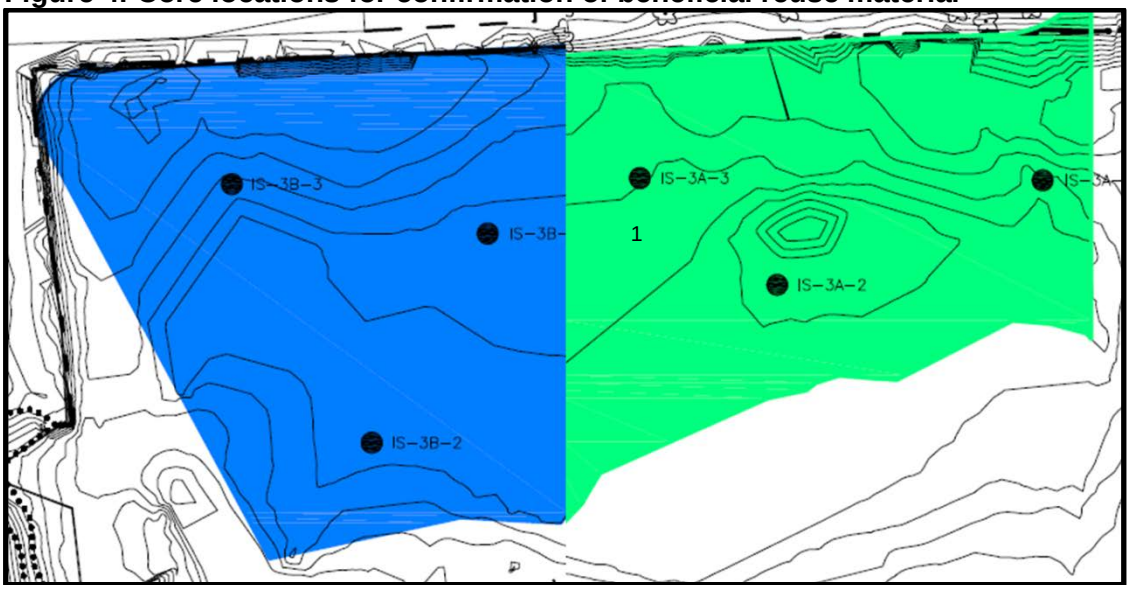
1. Beneficial Reuse – in situ cores prior to dredge activities – confirms material is suitable for placement in the habitat restoration area
2. Navigational spoils – pile sampling – confirms sediment is appropriate for Lot 24 placement
3. Environmental spoils – pile sampling – landfill specified acceptance testing (Note:see Appendix E for the landfill waiver of the physical testing requirements)
4. Confirmation – in situ surface samples – confirms sediment quality is acceptable to remain in the harbor

Beneficial Reuse Sampling
Sevenson will perform six vibracore samples to meet the contractual requirements of in-situ sampling and testing confirmation for the beneficial reuse dredged material. The region selected by Sevenson for beneficial reuse material contains the largest percentage of granular materials by weight, as provided in the sampling results with the bid information. Sevenson anticipates that the transition between material types to be very apparent based on this information. The six samples will be performed in two distinct areas:

1) Dredging Task 3a and
2) Dredging Task 3b.

See locations on Figure 4 below.

Figure 4: Core locations for confirmation of beneficial reuse material



Based upon the contract documents (developed through past sampling events), the Task 3a samples do not have an overlying stratum of environmental (landfill) sediments. The Task 3b sample locations have 2 to 4 feet of overburden that must be stripped off prior to beneficial reuse. Locations, elevations and thicknesses for coring/sampling are detailed in the table below.

Table 4 Coring Locations with targeted core elevations and length

Sample ID	Northing	Easting	Surface Elevation* *ft NAVD88*	Target Depth *ft NAVD89*	Core Length *ft*	Over Burden Thickness* *ft*	Interface Elevation* *ft NAVD88*
IS-3a-1	146158.71	812683.47	575.0	568.0	7.0	0	575.0
IS-3a-2	146109.11	812557.58	578.5	568.0	10.5	0	578.5
IS-3a-3	146159.75	812492.44	575.0	568.0	7.0	0	575.0
IS-3b-1	146128.91	812418.65	576.5	568.0	8.5	3	573.5
IS-3b-2	146029.76	812363.74	577.0	568.0	9.0	4	573.0
IS-3b-3	146152.30	812297.43	575.0	568.0	7.0	2	573.0

** Based upon Contract Plans*

Either prior to sampling or coordinated with land personnel, Sevenson will obtain current water elevation data from the reference location. Sevenson will navigate to each location, measure the water depth, and collect the core as detailed in the provided SOP. The length of core targeted for sampling is at least six inches below the targeted removal elevation. In addition to recording the actual sample locational, field records will indicate the date and time of core collection and the depth of the core. After retrieving the core, Sevenson will mark and measure the core length from the top of the core to bottom to ensure 85% recovery was achieved. If 85% recovery is not achieved, a new core will be performed, up to two additional cores. Should the third core still not achieve 85%, the recovery percentage will be noted and composite samples will be collected from the obtained cores with preference given to the core with the greatest

recovery unless there are concerns that the core material is not representative of the deposit. Photographs of the collected core may be taken either by Sevenson or Robert E. Lee & Associates (REL) personnel.

Sevenson will collect all of the cores in the field prior to processing them. At Sevenson's discretion, one location may be selected for collection of a replicate core. Cores will be stored vertically and transported to the processing location on land. If cores are not processed in the same day of collection, they will be capped and stored vertically until processing. Refrigeration is not required.

Cores will either be processed in the on-site warehouse leased by Sevenson or within the confines of the dewatering pad. All processing work will occur over a tarp on a clean surface isolated from any potential contamination source. Prior to processing each core, the Sevenson sampler will note and mark any changes to the position of water-sediment interface. For cores collected from Task 3a locations where the full core is anticipated to contain beneficial use material, the water layer must be carefully removed from the top of the core to minimize disturbance on the sediment surface. For cores collected from the Task 3b locations, minimizing disturbance of surface sediments is less important. Any alternative sample handling procedures must be approved by the DNR project manager prior to implementation.

Based on visual assessment of the core and considering historic characterizations provided with contract information, the sampler will mark the core location below which material is expected to be beneficially reused. This mark will be measured from the top of sediment and used to refine the dredging plan. The visual assessment will consider color stratification, material types, and grain size to identify the material for beneficial reuse. Sediment below the marked location will be segmented into no greater than two foot increments (based on NR 347.06 Wis. Adm. Code requirements) with the beginning and ending measurements of each segment recorded in field notes. Smaller segments may be designated based on visual assessment of the core and project needs. REL will photograph core segments. Representative material from each segment will be sampled, homogenized and then sub-sampled for metals analyses. A sub-sample for particle size will be collected and archived in the event that this data is necessary for decision-making. Each segment will be uniquely labeled with the core identity (see Table 1) and segment letter designation. Corresponding depth interval information will be recorded in field notes and provided to REL and DNR. All sediment and trash resulting from the coring will be disposed of at the WM landfill.

At a minimum, the top 2 feet of material sampled will be submitted for analysis for target metals**.** One core in area 3b will be selected for field duplication of the top sampled segment. (Note: the location was adjusted to 3a based on field conditions.) Remaining samples from lower segments may be archived in the event that analysis of the targeted interval exceeds the TEC decision threshold. Chain of custody documentation will be completed and samples will be packaged for delivery to the laboratory. Sediment samples for metals analyses (and associated moisture) may be stored at room temperature prior to shipment to the laboratory.

Dredge Management Units Sampling Overview

Sevenson's addendum to the Work and Dewatering Plan (Appendix B) designates a series of seven tasks in six areas or zones based on the work to be accomplished and the fate of the material to be dredged. The maps of the dredge task areas presented here were updated on August 29, 2014 after a project planning meeting. Some of the tasks overlap geographic areas (e.g. Dredging Task 3b and 4a are in the same area and Dredging Task 5 encompasses all of the area in Dredging Task 4b plus additional area.) The sediment sampling design is based on

the dredging workplan, the site conceptual model, and project decisions or assessments associated with each area.

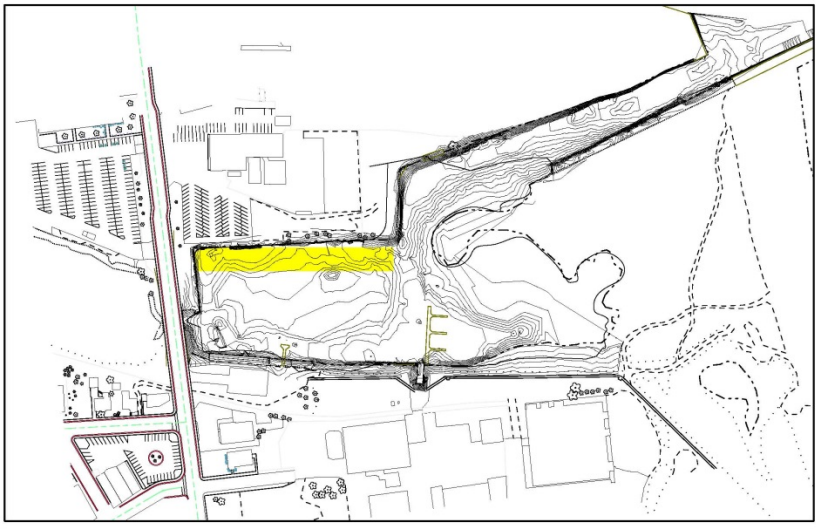
Table 5: Summary of In-harbor Sampling by Management Unit/Dredge Task

Management Unit	Dredge Task	Verification Sampling (Dredge Task)	Decisions
1	1	None	None – Operational unit
2	2	• Post-dredge bathymetry • Confirmation Sampling within one foot of target placement Post-placement bathymetry • Final Confirmation Sampling	• Dredge design elevation reached • Placed sediments below TEC, verification of conceptual site model, operational adjustments for final foot of placed sediment to assure acceptable surface concentrations • Habitat placement elevations acceptable • Surface sediments below TEC
3	3A	Post-dredge bathymetry	Navigational elevations below 568.0 msl, acceptable slopes
4	3B/4A	• Post-dredge bathymetry (3B) • Post-dredge confirmation sampling (3B) • Post-dredge bathymetry (4A)	• Dredge design elevation reached • Environmental spoils removed acceptably and OK to proceed with placement in DMU 2 • Navigational elevations below 568.0 msl, acceptable slopes
5	4B	• Post-dredge bathymetry • Post-dredge confirmation sampling (hatched area only)	• Dredge design elevation reached • Surface sediments below TEC
6	5	Post-dredge bathymetry	Navigational elevations below 568.0 msl, acceptable slopes

The management units, associated dredge tasks and sampling design elements are as follows:

Management Unit 1 – Dredge Task 1

Figure 5: Management Unit 1

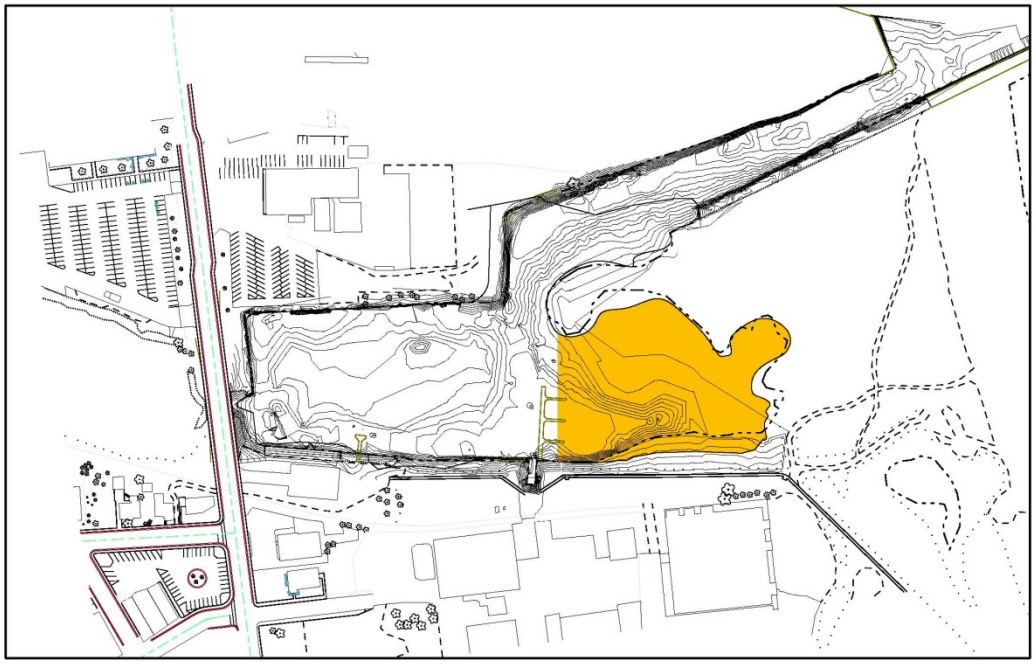


DREDGE TASK 1

This management unit is adjacent to the sediment loading area and work will focus on preparing the land-based unloading area. No sampling will be associated with this unit.

Management Unit 2 – Dredge Task 2 – Habitat Restoration Area

Figure 6: Management Unit 2



Based on historic sediment characterization, the sediments targeted for dredging in management unit 2 include material for landfill disposal and clean material that can be placed in Lot 24. Environmental dredge spoils and navigational dredge spoils will be placed in barges and then unloaded into appropriate pad for stockpiles in the designated work area. Stockpiled dredge spoils will be sampled as described in subsequent sections. Following dredging to the design elevation, REL will perform a bathymetric survey as confirmation that dredging is complete. This dredge elevation is designed to over-dredge contaminated sediment and the area will be covered with clean sediment associated with habitat creation so confirmation sampling is planned upon dredge completion.

Sediments identified as appropriate for beneficial reuse will be removed from targeted locations and placed via hydraulic dredge into this area. When sediment placement is within approximately one foot of target elevations, REL will take confirmation samples of the surface sediments (top six inches) assessment of contaminant levels. Once Sevenson believes material placement achieves design elevations, REL will confirm the surface elevations meet specifications before proceeding to final confirmation sampling.

Management Unit 3 – Dredge Tasks 3A

Historic sediment characterizations in this area indicate that the material is appropriate for beneficial reuse in the Management Unit 2, with no overburden of contaminated sediments. As described in the beneficial use sampling above, this characterization will be confirmed by analysis of three core samples (consistent with technical specification 35 20 13 identifying a sampling frequency of 1 sample per 5,000 cubic yards).The actual material targeted for

beneficial reuse may be refined by the results of the core sampling.

Figure 7: Management Unit 3

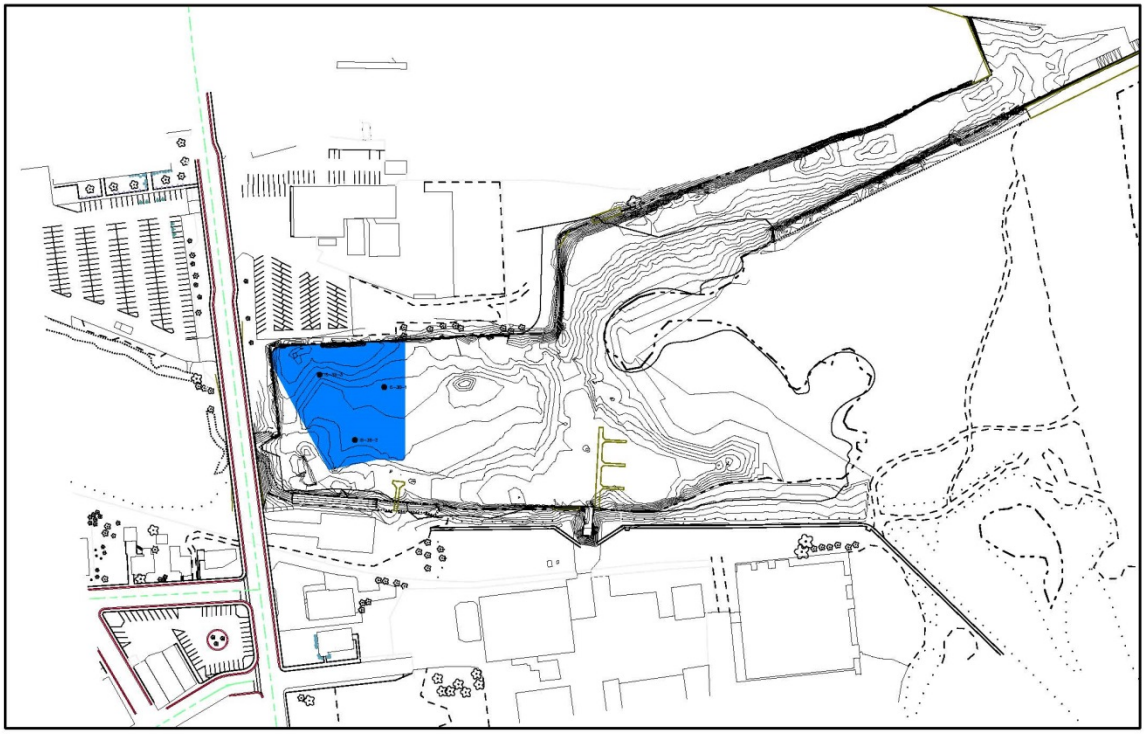


Material from Management Unit 3 will be hydraulically dredged and placed directly in Management Unit 2. Sediments in Management Units 4 and 5 to the west and south respectively contain environmental spoils so the dredging process will be managed to minimize the risk of slumping into this Management Unit and contaminating clean material. When Sevenson indicates that design elevations have been met, maximum elevation of 568.0 mean sea level (MSL) in the navigation area, REL will perform bathymetry to confirm the specifications have been met. (see assessment section for special considerations).

Management Unit 4 – Dredge Tasks 3B/4A

Historic sediment characterizations in this area indicate that the contaminated sediments overlie material that is appropriate for beneficial reuse in the Management Unit 2. As described in the beneficial use sampling above, this characterization will be confirmed by analysis of three core samples (consistent with technical specification 35 20 13 identifying a sampling frequency of 1 sample per 5,000 cubic yards).The dredge elevation for removing environmental spoils will be refined based on evaluation of the sediment core results.

Figure 8: Management Unit 4 – Task 3B

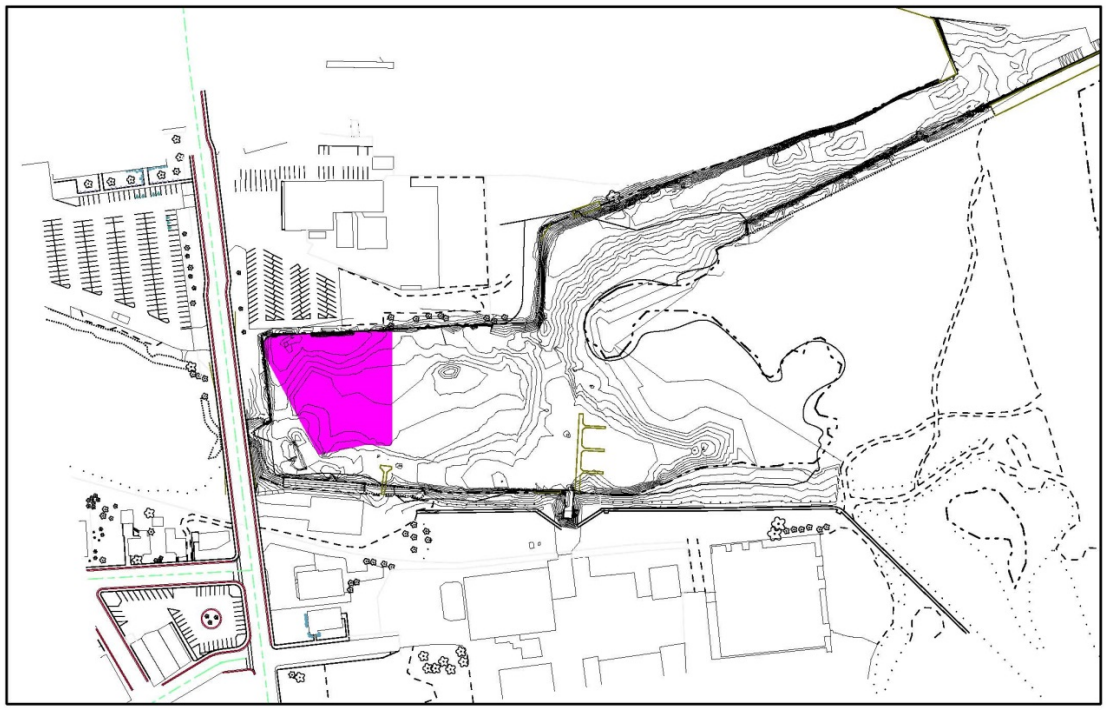


Sevenson identified mechanical dredging for Dredge Task 3B removal of environmental spoils to a barge and off-loading to the appropriate stockpile. Once Sevenson identifies the project team that Dredge Task 3B is complete, REL will perform bathymetry to confirm that target elevations have been met. If bathymetry confirms that specified removal elevation has been reached (see assessment section), then REL will perform confirmation sampling. If results of confirmation sampling indicate that contaminated sediments remain in the Management Unit, then additional dredging will be required followed by confirmation sampling in the re-dredged areas (see assessment section).

Once the REL and WDNR agree that environmental spoils have been removed acceptably, Sevenson will proceed to Dredge Task 4A, hydraulic dredging of navigational spoils and placement into Management Unit 2. Note that the plan to beneficially reuse these sediments may be adapted based on the results of the core sampling described above.

Following completion of Dredge Task 4A, REL will perform a bathymetric survey of the area to confirm that navigational elevations have been reached with stable side slopes. No confirmation sampling is required at the completion of this Dredge Task.

Figure 9: Management Unit 4 – Task 4A



Management Unit 5 – Dredge Task 4B

The material targeted for removal in Dredge Task 4B is environmental spoils for landfill disposal. With the exception of the hatched area adjacent to the piers (Harbor Town dock), the contaminated sediments overlie clean sediments that will also be removed. The hatched area contains contaminated sediments to approximately the removal elevation. Upon completion of dredging in Management Unit 5, REL will confirm that design elevations for contaminant removal have been reached across the full area. Following confirmation of acceptable elevations, REL will perform confirmation sampling within the hatched area. Remaining sediments outside of the hatched area will be removed from the harbor in a subsequent task so no confirmation sampling will be performed within the management unit.

Figure 10: Management Unit 5



Management Unit 6 – Dredge Task 5

Historic characterizations indicate that sediments within Management unit 6 are clean navigational spoils that are appropriate for placement in Lot 24. This includes the sediments below Management Unit 5 and into the channel to the north.

Figure 11: Management Unit 6



Upon completion of mechanical dredging in this area, REL will confirm that navigational and design elevations have been achieved. No confirmation sampling is required.

Confirmation Sampling

Confirmation sampling is planned for areas where there is a potential for contaminated sediments to remain in the harbor and is done in combination with visual assessment of dredged material and bathymetry confirming acceptable elevations and dredge surfaces have been reached.

Bathymetry

Initial baseline, bi-weekly, and final bathymetric surveys will be completed by Robert E. Lee survey crews. Survey data will be gathered (in NAVD88 DATUM) at a frequency necessary to adequately profile the existing grade within the harbor. Survey data will be completed using a SonarMite echo sounder and spot checked with traditional methods to verify accuracy. An initial survey will be conducted to develop a baseline bathymetric survey.

Bi-weekly checks will be made to validate progress payments of material removed by dredging. Bi-weekly checks will also be correlated to cross check depth and accuracy of dredge prior to conducting post-dredge confirmation sampling. Dredge depth accuracy will be obtained through the use of the HyPack Dredgepack dredge positioning software with DGPS antenna. (*Sevenson Work and Dewatering Plan – Appendix B*) with zone dredging completion and respective 24-hour post dredge sampling criteria.

Surveys will also be taken before and after bulk removal, then again after final placement of beneficial reuse material in the restoration area. One post-dredge, digital bathymetric survey shall be necessary for purposes of confirming final depths and contours. The design of these surveys shall follow the methodology established during the pre-dredge bathymetric survey performed by the design engineer. *See specification section 35 20 13 (Appendix C – Technical Specifications) and SOPs for REL* .

Topography

Surveys shall be necessary at both the harbor site and the Lot 24 containment site for purposes of preparing as-constructed site plans and as-constructed final grades site plans. These surveys shall follow established land survey techniques, and shall occur following completion of site preparation and following the conclusion of site operations and closure.

Table 6. Proposed Sampling Parameters

Sample Matrix	Sample Type	Location	Schedule	No. of Samples[1]	*Field Duplicates	Parameters
Sediment	Beneficial Use	In-situ – DMU 3 and 4	1 sample per 5000 cubic yards (3 cores each in DMUs 3 and 4)	12	1	Arsenic, copper, lead, mercury, zinc
	Navigational Spoils – representative grab	Stockpile at dewatering pad adjacent to dredging site	1 sample per 5000 cubic yards	5	1	Arsenic, copper, lead, mercury, zinc
	Confirmation – grab of top 6 inches of core (push core or ponar)	In-situ – DMU 4, 5 (sub-area) and post-placement in DMU 2	24 hours following removal of contaminated sediment from an area, at a rate of 1 sample per 2500 square feet of contaminated sediment remaining in harbor as described in section B2.2.2.	50[2]	5	Arsenic, copper, lead, mercury, zinc
	Environmental Spoils – representative grab	Stockpile at dewatering pad adjacent to dredging site	Monitoring waived by landfill (initially specified as 1 sample per 5000 cubic yards)	-	-	% solids/moisture; grain size; liquid limit, plastic limit, plasticity index, hydraulic conductivity

Wastewater	WWTP Compliance – grab sampling	Influent sample port at entrance to first influent equalization tank	1. Prior to initial discharge; 2. Two times on two different days during the first calendar week of discharge; 3. Once per week during the second through fifth weeks of discharge; 4. Once per month after the fifth week for the remaining duration of the project.	11	2	Arsenic, cadmium, chromium, copper, lead, mercury, nickel, selenium, silver, zinc, cyanide, oil & grease, phenols, pH, BOD, TSS, PHOS
		Effluent sample port at the bag filter effluent sample port	1. Prior to initial discharge; 2. Two times on two different days during the first calendar week of discharge; 3. Once per week during the second through fifth weeks of discharge; 4. Once per month after the fifth week for the remaining duration of the project.	11	2	Arsenic, cadmium, chromium, copper, lead, mercury, nickel, selenium, silver, zinc, cyanide, oil & grease, phenols, pH, BOD, TSS, PHOS
	Flow	After the bag filter unit and prior to the discharge point	Continuous, passive data collection	--	--	Flow

[1] Estimated based upon calculated volumes of sediment, calculated areas for confirmation sampling, core segmenting and estimated project length for WWTP discharge.
[2] Actual number of confirmation samples will vary based on final square footage of sampling areas.

B2. Sampling Methods

B.2.1 Interstitial Water

Grab samples will be utilized to sample influent and effluent flows. Influent samples will be taken at the entrance to the first influent equalization tank. Effluent samples will be taken at the bag filter effluent sample port. Effluent sampling requirements, methods, standards, and processes are outlined for both confirmation and compliance sampling by firms conducting these activities. *See Appendix D – Wastewater Treatment System Monitoring Plan and Appendix F – Standard Operating Procedures (Sevenson and Robert E. Lee)*.

B.2.2 Sediment

Sampling procedures implemented will be selected based on purpose for the samples and environmental conditions encountered. SOPs for Sevenson and REL are found in Appendix F.

B.2.2.1 Beneficial Use Samples

Planned sampling includes using a core to collect sediment samples to dredge depth. The area is known to contain woody debris so planned locations and sampling techniques may need adjustment to obtain representative samples. Cores will be assessed visually for strata and transition between any silty overburden and higher grain size materials. From that interface, cores will be segmented in no greater than 2 foot increments (per requirements in NR 347 Wis. Adm. Code). Segments will be composited and a subsample retrieved for chemical analysis. See section B.1.1 for additional information about core sampling.

B.2.2.2 Confirmation Samples (DMUs 4, 5, and 2)

In DMUs 4 and 5, confirmation sampling occurs once overburden is removed and a combination of bathymetry and visual assessment of dredge spoils confirms that target elevations for contaminated sediment removal have been met. In that area, REL will overlay an approximate 50’ x 50’ grid. The grid may be adjusted larger or smaller to account for edge and side slope areas. Within each gridded area, a sample location will be selected randomly. The preferred sampling method is to use a push core to retrieve 18 – 24”. Minimum acceptable retrieval is 6”. Any retrieved cores will be photographed. If the coring technique is unsuccessful in retrieving the minimum amount of material, the next choice of method for surface sampling is using a Ponar. If Ponar grab sampling is unsuccessful in retrieving a representative surface sample, REL will consult with WDNR and the project team to choose an alternative approach.

Cores will be capped and stored upright until processing on land. Prior to processing each core, the REL sampler will mark the position of water-sediment interface. The water layer must be carefully removed from the top of the core to minimize disturbance on the sediment surface. This may be accomplished by drilling a hole in the core tube above the interface and allowing the water to drain. If the surface sediments contain minimal silt or other small grain size material, it may be feasible to carefully decant the water. A representative sub-sample of the top 6” segment will be transferred to an aluminum pan, homogenized, and a sub-sample placed into a sample container for analysis immediately. The 6 – 12” segment and potentially the 12 – 18” may be subsampled using the same procedures described and then retained (archived) in the event surface sediment concentrations are above the TEC and additional characterization is necessary. If Ponar sampling is used, the entire grab sample will be homogenized and subsampled for subsequent analysis.

In DMU 2, sampling efforts must be coordinated with the hydraulic dredging placement activities to minimize effects on the schedule. Sampling will be limited to the surface material so the first choice method is with a Ponar. When placed material is within approximately 1 foot of the target elevation, the area or sub-area will be divided into approximately 100' x 100' grids. In consultation with WDNR, REL will select three locations within each gridded area and collect Ponar grabs from each station. Ultimately, the three grab samples will be homogenized and roughly equal aliquots (volumes) will be combined into a single composite that is thoroughly mixed prior to placement into a sample container for laboratory analysis. If any of the individual grabs appears to contain more silt than expected in the beneficial reuse material or appears intermingled with overburden, a separate subsample may be taken. The project team should be notified and the situation assessed (See section C). Once material is placed to the design elevation, REL will follow a similar procedure to collect samples from the final placed surface.

B.2.3 Bathymetry

Hydrographic surveying shall be conducted via conventional surveying methods that meet the requirements of the Corps of Engineers Engineering Manual EM-1110-2-1003 (2002) Soft Bottom Dredging Support Survey (±0.2 feet vertical and ±2 feet horizontal accuracy). Robert E. Lee will conduct the bathymetric surveys using a SonarMite echo sounder, and will conduct spot checks with traditional methods to verify the accuracy of the equipment. *See Appendix F – Standard Operating Procedures (Sevenson and Robert E. Lee) and COE Manual EM-1110-2-1003* hydrographic survey protocol.

B.2.4 Topography

Post-construction topographic surveys shall be conducted via conventional surveying methods that meet the requirements of Wisconsin Administrative Code Chapter A-E 7*. See Appendix F – Standard Operating Procedures (Sevenson and Robert E. Lee*).

B3. Sample Handling & Custody

B3.1 Sample Collection Documentation

Sample-handling procedures include field documentation, COC documentation, sample shipment, and laboratory sample tracking. Various aspects of sample handling and shipment, as well as the proposed sample identification system and documentation, are discussed in the sections below.

B3.1.1 Field Books

Records of the field activities will be maintained in field books dedicated to the City of Marinette Project. Entries will be dated and signed by personnel recording the data. The entries will be made in ink. At a minimum, information recorded in the field books will include documentation of sample locations, sampling times, types of samples collected, weather conditions, and any other information pertinent to the assessment. For sediment core samples, entries will include core recovery and interval composited (e.g. 2 – 4') associated with the field identifier.

Any photographs taken will be recorded in the field book or on a photo log. In addition to the picture number and subject matter, the identity of the photographer should also be noted. If the camera does not store locational information and date and time, the logged entry should include

this information.

B3.1.2 Field Identification System

Each sample collected during property assessments will be given a unique identification code. Each unique sample identification will consist of the following:

	Field Identification Label	Label Summary
	Sediment Testing	
Contractor	IS-(area*)-(sequential numbering)+ letter**	In-Situ testing - Material to Beneficial Reuse
	NSS - (area*)-(sequential numbering)	Navigational Stockpile Sample - material to Lot 24
	ESS - (area*)-(sequential numbering)	Environmental Spoils Stockpile Sample - Material to Landfill
REL	LCS - (area*)-(sequential numbering)	Landfill determination - Beneficial Reuse - Confirmation Grab Sample
	FCS - (area*)-(sequential numbering)**	Final Dredge - Confirmation Grab Sample
	BCS - (area*)-(sequential numbering)	Confirmation Sample in post placement Beneficial Reuse Area
	Wastewater Testing	
Contractor	WTS - (IN or EF) - (sequential numbering)	Influent (IN) or Effluent (EF) WWTP Compliance Testing

* Area" referenced per dredging task figure in Sevenson's Supplement to work and Dewatering Plan, Appendix B

** lettering will be used starting with "a" and will be used alphabetically for each 2 foot sample of beneficial reuse material from the top of beneficial reuse material down to the bottom of the template. If core segments are samples collected by REL, the field number will include the sampling interval (e.g. 0 – 6").

B1.3.1.3 Field Sample Handling

Sample bottle labels appropriate for the size and type of containers shall be provided by the Laboratory. The sample containers will be labeled at the time of sample collection but prior to being filled. Each label will indicate at a minimum:

- Sample identification
- Date/time of sample collection
- Sampler's initials
- Required analyses
- Type of preservative

All labels will be completed in waterproof ink.

Possession and handling of samples will be documented from the time of collection to delivery to the laboratory. Sevenson/REL field personnel are responsible for ensuring that COC procedures are followed. Field personnel will maintain custody of all samples until they are relinquished to another custodian, the laboratory, or to the freight shipper. All samples must be catalogued on a COC form using sample identification codes. The date and time of collection

will be recorded on the form, as well as the number of each type of sample, the method of preservation, and the type of analysis. The COC SOP is located in *Appendix E*.

B1.3.1.4 Field Sample Packaging and Shipping

Samples will be packaged and transported in a manner that maintains the integrity of the sample and permits the analysis to be performed within the prescribed holding time. Prior to shipment, each sample container will be inspected for a label with the proper sample identification code.

Samples will be either couriered or shipped via overnight mail to the appropriate laboratory. The laboratory will be contacted in advance to expect shipment so that holding times of the samples will be conserved. The COC forms will be sealed in a plastic bag and transported inside the sample cooler. In addition, any shipping receipts will be incorporated into the COC documentation. Samples will be packed in the cooler using bubble-wrap packing materials and ice will be sealed in a Ziploc®-type bag. Any samples suspected of being highly contaminated will additionally be sealed in a Ziploc®-type bag. The cooler will be taped closed using two custody seals provided by the laboratory to prevent tampering during transport. Upon relinquishing the sample cooler to the laboratory, Sevenson/REL field personnel will sign custody of the samples over to the laboratory by signing and dating the bottom of the COC form. One copy of the COC documentation will be retained by the Sevenson data manager and a second copy will be retained by the laboratory. The integrity of the custody seals shall be noted by the laboratory on the COC form upon arrival. In addition, the shipping label will be included with the COC form retained by the Sevenson data manager.

B1.3.1.5 Field Documentation

Field COC procedures will ensure the proper documentation of each sample from collection in the field to delivery at the laboratory. Custody of samples shall be maintained and documented at all times. The documentation for each sample will include the following information:

- COC form
- Sample label with sample identification code
- Shipping documents.

This documentation will allow for proper identification and verification of all samples upon arrival at the laboratory.

B3.1 Sample Containers, Preservation, Packaging and Shipping

The number and type of sample containers, volumes, and preservatives are listed in *Table 7*. The containers are pre-cleaned and will not be rinsed prior to sample collection. Preservatives, if required, will be added by Sevenson/REL to the containers prior to shipment of the samples to the laboratory

All sample containers supplied by the laboratories will be cleaned according to U.S. EPA standards. QC documentation will be supplied with the sample containers and preservatives in order to verify their purity. The containers and preservatives can be traced back to their certificate of analysis from their lot number. The QC documentation/certificate of analysis shall be maintained on file with the analytical laboratories. Additionally, Pace shall provide the field team laboratory-grade deionized (DI) water for rinsing field equipment and instruments

B3.1.1 Water Samples

Water samples that are preserved by adding acid to the sample bottle will be capped and lightly shaken to mix in the acid. A small quantity of sample will be poured into the bottle cap where the pH will be measured using pH paper. The pH must be $\leq$2. Water samples that are preserved by adding sodium hydroxide should have a pH >12.

Table 7: Sample Container, Preservation and Holding Time Requirements

Matrix	Analytes	Bottles	Preservatives	Holding Time
Sediment	Metals (As, Cu, Pb, Zn)	1 x 4 oz. plastic jar	Unpreserved	6 months
	Metals (Hg)			28 days
	Sieve	1 x 16 oz. plastic jar	Unpreserved	N/A
	Hydrometer	1 x 16 oz. plastic jar	Unpreserved	N/A
	Atterberg Limits	1 x 16 oz. plastic jar	Unpreserved	N/A
	Hydraulic Conductivity	1 x gallon freezer bag	Unpreserved	N/A
	% Moisture	1 x 4 oz. plastic jar	Unpreserved	7 days
Interstitial Water	Metals, mercury	1 x 250 ml plastic jar	Nitric Acid (HNO_3)	28 days
	Metals, other	1 x 250 ml plastic jar	Nitric Acid (HNO_3)	6 months
	Cyanide	1 x 250 ml plastic jar	Sodium Hydroxide (NaOH)	14 days
	Oil & grease	1 x 1000 ml glass jar	Hydrochloric Acid (HCl)	28 days
	Phenols	1 x 1000 ml glass jar	Sulfuric Acid (H_2SO_4)	28 days
	pH	1 x 250 ml plastic jar	Unpreserved	Immediate
	BOD TSS	1 x 1000 ml plastic jar	Unpreserved (Iced)	48 hours
	Phosphorus	1 x 1000 ml plastic jar	Unpreserved (Iced)	7 days
		1 x 250 ml plastic jar	Sulfuric Acid ($H_2 SO_4$)	28 days

B3.2 Packaging and Shipping

All sample containers will be placed in a strong-outside shipping container. The following outlines the packaging procedures that will be followed for low concentration samples.

1. When ice is used (necessary for water samples only), pack it in zip-lock, double plastic bags. Seal the drain plug of the cooler with fiberglass tape to prevent melting ice from leaking out of the cooler.
2. The bottom of the cooler should be lined with bubble wrap to prevent breakage during shipment.
3. Check screw caps for tightness and, if not full, mark the sample volume level of liquid samples on the outside of the sample bottles with indelible ink.
4. Secure bottle/container tops, preferably with tape.
5. Affix sample labels onto the containers with clear tape.
6. Wrap all glass sample containers in bubble wrap to prevent breakage.
7. Seal all sample containers in heavy duty plastic zip-lock bags. Write the sample numbers on the outside of the plastic bags with indelible ink.

8. Place samples in a sturdy cooler(s) lined with a large plastic trash bag. Enclose the appropriate COC(s) in a zip-lock plastic bag affixed to the underside of the cooler lid.
9. Fill empty space in the cooler with bubble wrap or Styrofoam peanuts to prevent movement and breakage during shipment. Vermiculite should also be placed in the cooler to absorb spills if they occur.
10. If shipping water samples, ice used to cool samples will be double sealed in two zip lock plastic bags and placed on top and around the samples to chill them to the correct temperature.
11. Each ice chest will be securely taped shut with fiberglass strapping tape.
12. WALTCO Inc., 112 Packerland Drive, Green Bay, WI is the expected courier.

B4. Analytical Methods

Laboratory methods, detection limits, and protocol are listed in *Appendix I (Table 5)*. Methods selected for these samples must meet laboratory certification requirements of NR 149 Wis. Adm. Code.

Bathymetry

Data collected and processed through Hypack Survey Version 11.0.1.49 (or similar) software shall then go through the quality control methodology prescribed in B5 using Hypack Single Beam Editor software. This data can be plotted and analyzed in AutoCAD Civil 3D. Final dredge volumes shall be calculated by comparison of the post-dredge surveyed 3D surface and the pre-dredge surveyed 3D surface. See *Appendix F – Standard Operating Procedures (Sevenson and Robert E. Lee)*.

Topography

See Appendix F – Standard Operating Procedures (Sevenson and Robert E. Lee).

B5. Quality Control

Interstitial water and sediment samples will be handled in accordance with the processes outlined below. Additional supplementary information is provided in *Appendix D – Wastewater Treatment System Monitoring Plan, Appendix F – Standard Operating Procedures (Sevenson and Robert E. Lee), and Appendix G Table 6, and – Quality Assurance Manual and Standard Operating Procedures (Pace Analytical)*.

B5.1 Field Quality Control Samples

Where applicable, QC checks will be strictly followed during the investigation through the use of replicate measurements, equipment calibration checks, and data verification by Sevenson field personnel and the REL QA manager. Field sampling precision and data quality will be evaluated through the use of sample duplicates, equipment blanks, and field blanks. Sample duplicates provide precision information regarding homogeneity, handling, transportation, storage, and analyses. Equipment (rinsate) blanks will be used to assure that proper decontamination procedures have been performed and that no cross-contamination has occurred during sampling or transportation. Field blanks will be collected to detect possible sample contamination from ambient conditions in the sample area. If there is any discrepancy in the sample data, the Sevenson Project Manager will be notified and resampling of the questionable point scheduled, if deemed necessary. Requirements for field QA/QC samples are listed in Table 6. Decisions as to where and when the blanks will be sampled will be made in the field.

B5.1.1 Equipment Blanks

Equipment rinsate blanks will be collected to evaluate field sampling and decontamination procedures of non-dedicated equipment by pouring deionized water over the decontaminated sampling equipment. One equipment rinsate blank will be collected per matrix each day that sampling equipment is decontaminated in the field. Equipment rinsate blanks will be obtained by passing water through or over the decontaminated sampling devices used that day. The rinsate blanks that are collected will be analyzed for metals.

The equipment rinsate blanks will be preserved, packaged, and sealed in the manner described for the environmental samples. A separate sample number and station number will be assigned to each sample, and it will be submitted blind to the laboratory.

B5.1.2 Temperature Blanks

For each cooler that is shipped or transported to an analytical laboratory a 50-ml plastic container will be included that is marked “temperature blank.” This blank will be used by the sample custodian to check the temperature of samples upon receipt.

B5.1.4 Assessment of Field Variability

Duplicate sediment or water samples will be collected at sample locations to be determined in the field based on sample location observations, historical information, or field screening results (e.g. samples from these locations are expected to exhibit moderate or high concentrations of contaminants).

Sediment samples will be homogenized with a trowel in a sample-dedicated 1-gallon disposable container. Homogenized material from the container will then be transferred to the appropriate containers for both the regular and duplicate samples.

When collecting duplicate water samples, bottles with the two different sample identification numbers will alternate in the filling sequence. Note that bottles for one type of analysis will be filled before bottles for the next analysis are filled.

Duplicate samples will be preserved, packaged, and sealed in the same manner as other samples of the same matrix. A separate sample number and station number will be assigned to each duplicate, and it will be submitted blind to the laboratory.

B5.2 Laboratory Quality Control Samples

The laboratory QA manager will be responsible for ensuring that the laboratory’s data precision and accuracy are maintained in accordance with method and laboratory certification requirements.

B6. Instrument/Equipment Testing, Inspection, and Maintenance

B6.1 Field Screening Instruments

Sevenson’s field technical staff members will audit and maintain the performance field-screening instruments. Instruments will be calibrated, when applicable, according to the standard procedures. Regular preventative maintenance will be performed on a daily basis. See Sevenson SOPs in *Appendix F.*

Bathymetry
In order to test function of GPS receiver, follow methodology in B5. No testing, inspection, or maintenance is necessary for the survey pole or lead line.

B6.2 Decontamination Procedures

The decontamination procedures that will be followed are in accordance with approved procedures. Decontamination of sampling equipment must be conducted consistently as to assure the quality of samples collected. All equipment that comes into contact with potentially contaminated soil or water will be decontaminated. Disposable equipment intended for one-time use will not be decontaminated, but will be packaged for appropriate disposal. Decontamination will occur prior to and after each use of a piece of equipment. All sampling devices used will be steam-cleaned or decontaminated according to the following procedure.

- Non-phosphate detergent and tap water wash, using a brush if necessary
- Tap-water rinse
- Deionized/distilled water rinse
- Deionized/distilled water rinse (twice)

Equipment will be decontaminated in a pre-designated area on pallets or plastic sheeting, and clean bulky equipment will be stored on plastic sheeting in uncontaminated areas. Cleaned small equipment will be stored in plastic bags. Materials to be stored more than a few hours will also be covered.

Boats and other in-water equipment must be cleaned in accordance with DNR procedures for the control of spread of invasive species.

B7. Instrument/Equipment Calibration and Frequency

B7.1 Instrument Calibration and Frequency

The responsibility for the calibration of laboratory equipment rests with Pace Laboratory. Sevenson and REL field personnel are responsible for the calibration of their respective field equipment and field equipment provided by subcontractors.

Documented and approved procedures will be used for calibrating measuring and testing equipment. Widely accepted procedures, such as those published by U.S. EPA and American Society for Testing and Materials (ASTM), or procedures provided by manufacturers in equipment manuals will be adopted.

Equipment that fails calibration or becomes inoperable during use will be removed from service, segregated to prevent inadvertent use, and tagged to indicate the fault. Such equipment will be recalibrated and repaired to the satisfaction of the laboratory personnel or Sevenson/REL field personnel, as applicable. Equipment that cannot be repaired will be replaced.

Records will be prepared and maintained for each piece of calibrated measuring and test equipment to document that established calibration procedures have been followed. Records for subcontractor field equipment and Sevenson/REL equipment used only for this specific project will be kept in the project files. Pace Laboratories will maintain laboratory calibration records.

7.2 Field Instrument Calibration

Instruments used to gather, generate, or measure field environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specification. As applicable, field instruments will be calibrated daily prior to use and the calibration will be verified by analyzing a calibration check standard. The calibration will be consistent with the standard procedure.

- Seametrics flow meter: The field calibration procedures are presented in the field SOPs located in Appendix E for the Seametrics flow meter. The meter is factory calibrated and in the field calibration is not typically required.
- Sonarmite Echo Sounder: Field calibration procedures are presented in the REL SOP in *Appendix G*
- Trimble R8 GNSS System:

Calibration procedures will be documented in the field logbook and field sampling sheets. Documentation will include the following:

- Date and time of calibration
- Identity of the person performing the calibration
- Reference standard used, if applicable
- Reading taken and adjustments to attain proper reading
- Any corrective action

Trained personnel will operate field measurement equipment in accordance with the appropriate standard procedures or manufacturer's specifications. Field technical staff members will examine field measurement equipment used during field sampling to verify that they are in operating condition. The Sevenson field team leader will periodically audit the calibration and field performance of the field equipment to ensure that the system of field calibration meets the manufacturer's specifications.

Bathymetry and Topography
A control point of known coordinates shall be surveyed as a ground control check of the accuracy of the GPS receiver. A USACE control point is located on the Ogden Bridge at the west side of the project area and is a good option. This control point is located at 812178.28 feet East, 145998.65 feet North, 589.06 feet Z (Wisconsin County Coordinate System, Marinette Zone, North American Datum of 1983, adjustment 1991). The GPS receiver shall also use a cellular modem capable of receiving real-time corrections from the Wisconsin Continuously Operating Reference Stations (WISCORS).

7.3 Laboratory Instrument Calibration

The calibration procedures and frequencies of the equipment used to perform the analyses will be in accordance with requirements established by the method and Wisconsin Laboratory Certification requirements Laboratory quality control, calibration procedures, and corrective action procedures are discussed in the Pace Laboratory's QA Manuals. Pace Laboratory's QA Manual is located *in Appendix G*.

B8. Inspection/Acceptance of Supplies & Consumables

Shoreline treatments shall be repaired or replaced incidental to dredging activities. Sevenson has two subcontractors (MJB & UP Engineers & Architects) who will be addressing the technical

requirements for concrete and shoreline installation and testing. UPEA will be handling concrete bearing capacity testing and Proctor and compaction testing for shoreline stability. Refer to *Appendix C* for technical specifications and Sevenson certifications for subcontractors with regard to concrete and Proctor testing protocol.

U.P. Engineers and Architects are providing Aggregate Testing, concrete testing, and density testing to accommodate the requirements of the project. ASTM standards will be used for all methods and the certification and ASTM processes.

B9. Data Acquisition Requirements for Non-Direct Measurements

Not applicable.

B10. Data Management

B10.1 Data Management

Sevenson's field technical staff members and the REL oversight team will each manage raw data for which they are responsible during field activities. Data will be recorded on the appropriate field forms (examples of which are located in *Appendix F*) or in field logbooks. The Sevenson data manager will periodically collect data gathered during sampling activities in order to maintain results. As appropriate, the Sevenson data manager will coordinate transfer of raw data to computer formats such as Microsoft® Excel or Microsoft® Access to better organize and track incoming data. This will enable the Sevenson data manager to identify any data gaps.

The Pace Laboratory project manager will be responsible for laboratory data management. Pace Laboratory's procedures for data review and data reporting are discussed in Section 12.0 of their QA Manual, located *in Appendix G*. Analytical data reports generated by Pace Laboratory will present all sample results, including all QA/QC samples. The data reports will include a laboratory narrative for the data set describing any out of control analyses and their effect on sample results, explanation of all lab applied qualifiers; all sample results including the % moisture content for soil samples, the spike and duplicate analysis results (or MS/MDS results) including the % recoveries and RPDs.

The following data must be available upon request from the lab on a case by case basis, if data issues arise:

- summaries of daily calibration check samples (including notation of any outliers),
- calibration blank results,
- method blank results,
- lab control sample (LCS) results including % recoveries.

Sediment results will be reported on a dry weight basis. All data, including QA/QC results, will become part of the project files and will be maintained by the Sevenson data manager. Upon report delivery, Sevenson personnel will analyze laboratory data in accordance with accepted statistical methodologies and will be supervised by the Sevenson data manager.

B10.2 Bathymetry Data Management

Data will be both digital and written, and shall remain in the custody of the collector until it can be uploaded to a project server for analysis. Any written data shall be scanned and uploaded as

well. Folder names should be clearly labeled as to the data contained within. The data will then be shared digitally with the personnel performing analysis and/or QA/QC.

SECTION C – ASSESSMENT AND OVERSIGHT

C1. Assessments and Response Actions

C1.1 General Project Operations

On a daily basis during project activities construction oversight personnel shall evaluate performance of work and adherence to permit conditions and QAPP procedures as well as relay any issues to project management as they arise. Project management shall be responsible for providing recommendations for any deficiencies. The contractor shall be responsible for correcting deficiencies, which the oversight personnel shall verify and document.

On a weekly basis—and within 24 hours of a 24-hour rainfall event of 1/2 inch or greater—environmental compliance personnel shall inspect the Menekaunee Harbor and Lot 24 project sites for compliance to the WPDES storm water general permit. This inspector shall keep a record of the installation, maintenance, and state of erosion and sediment controls, identify deficiencies, and provide recommended corrective actions. The contractor(s) shall be responsible for correcting deficiencies within **one day** of their identification. The inspector shall verify executions of corrective actions and document them in the Erosion Control & Storm Water Management Plan per NR 216 and NR 151 Technical Standards.

On a weekly basis environmental compliance personnel shall inspect the Menekaunee Harbor and Lot 24 project sites for compliance to the following permits:

1. WDNR Chapter 30 Waterway Individual Permit for Dredging Operations
2. US Army Corp of Engineers Section 404/401 Permit for work in a waterway
3. Lot 24 Dredge Spoils Disposal Site Low Hazard Waste Exemption Request Approval.

This inspector shall keep a record of the installation, maintenance, and state of erosion and sediment controls, in-water TSS controls/BMPs (turbidity barriers), wastewater treatment processes, and general compliance. The inspector shall identify deficiencies and provide recommended corrective actions. The contractor/s shall be responsible for correcting deficiencies within **one day** of their identification.

Periodically during construction the project engineer shall perform inspections to verify proper installation and quantities associated with rip rap, concrete, rebar, and other construction materials. These audits will occur at critical times in construction while any deficiencies can still be easily addressed, as well as prior to fulfillment of contractor pay requests. If any deficiencies are identified during an audit the auditor shall be responsible for recommending corrective actions, and the contractor shall be responsible for addressing these corrective actions. If quantities are found to be different than those reported by the contractor than the corrective action taken out by the contractor shall be revising the pay request as appropriate.

C1.2 Bathymetry – Confirmation of target elevations

As a quality control measure, there shall be no areas that are left 4-inches or greater above design grades. In the event areas are left between 0”-4” above plan grade, the project area will be sectioned off into 50’ x 50’ grid sections. If the average depth over adjacent grid sections fall short of the dredging target depths, it will be expected the contractor continue to dredge the area to plan grades. If the average depth falls short of the target dredge depths in only one grid section, and do not exceed 4-inches, dredging activities would be considered complete and the

contractor would not be required to do additional dredging, unless they so desire to dredge to target depth.

C1.3 In-situ Sediment Core Sampling

If evaluation of core recoveries or core materials suggests that samples may not be representative of deposit materials or recoveries are below 50%, the sampling team may attempt sampling in alternate locations within the target area, adjust the sampling technique, or, in consultation with the project team, select another sampling method

C1.3.1 Confirmation Sampling

In DMU 4, if visual examination of sampled sediment indicates presence of fine grained material characteristic of environmental spoils, then additional sampling may be done to identify the extent of re-dredging necessary. Re-dredging decisions may also be based on a combination of visual examination and chemical testing when visual examination is inconclusive or potentially nominal amounts of environmental spoils are present. If visual examination indicates environmental spoils have been adequately removed, then beneficial use placement may begin before results for chemical analyses are available. If chemical testing subsequently indicates contaminant concentrations above the TEC, the project team will revisit assumptions in the conceptual site model and determine whether adjustments in operational procedures are necessary moving forward. Potential actions include but are not limited to collecting additional samples, adjusting the sampling plan, and evaluating locations targeted as containing beneficial use sediments. The team anticipates that sediments already placed in the beneficial use area will remain in DMU 2 (assuming that the contaminant concentrations are not above the MEC and additional sediments will be placed above it).

In DMU 5, if the results for surface samples are above the TECs below, additional dredging will be necessary. Visual examination may also be used to assess whether re-dreding is necessary. Archived sub-surface samples may be examined or analyzed and bathymetry re-evaluated to inform decisions about the extent of re-dredging to be performed.

TECs
9.8 mg/kg arsenic;
32 mg/kg copper;
36 mg/kg lead;
0.18 mg/kg mercury
120 mg/kg for zinc.

If confirmation samples following re-dredging are above TECs, the project team will do a more extensive evaluation of the potential causes. Consideration will be given to sample proximity to adjacent contaminated areas and additional sampling may be considered.

For surface sediment samples in DMU 2 collected at one foot before final elevation is reached, individual samples may be analyzed if atypical material is encountered or the composite sample results are above the TECs. Additional grab samples may be collected to determine the extent of the area affected. The project team will investigate potential sources of contaminated material and consider options for placement of the final foot of material to assure surface sediments are below TECs.

C1.4 Navigational Dredge Spoils

If arsenic concentrations in the navigational dredge spoils pile are > 6.0 mg/kg, the affected stockpile will be sent to the landfill for disposal. Sevenson will consider whether dredge procedures need to be modified to minimize the amount of material that fails the criteria for placement in Lot 24. Additional assessment of the sediments may be considered. If Sevenson anticipates that the amount of material disposed at the landfill exceeds projected amounts because navigational dredge spoils exceed Lot 24 placement criteria, Sevenson must notify REL and the city of Marinette in a timely manner and identify options that control costs and are consistent with project objectives. Selection among the options will be done in consultation with the project team. The city of Marinette has responsibility for authorizing any change orders.

C1.5 Visible Plumes Outside Turbidity Barriers

If visible sediment plumes are observed outside the turbidity barriers, Sevenson or REL may issue a stop work order until the cause can be identified. Sevenson will implement best management practices to control the plume in accordance with its Chapter 30 permit.

C2. Reports to Management

Construction oversight personnel shall provide weekly reports to project management. Project management shall be responsible for the construction contractor to relay recommendations and discuss any concerns. These reports shall be uploaded to the project FTP site and an email sent to involved parties to notify them of new reports and to highlight any critical information.

Environmental compliance personnel shall provide weekly stormwater compliance inspection reports to project management and the construction contractor. The construction contractor shall be responsible for corrective actions for any deficiencies identified within this report. These reports shall be uploaded to the project FTP site and an email sent to involved parties to notify them of new reports and to highlight any required corrective action.

Construction contractor management shall provide progress updates to project management at regularly scheduled meetings or teleconferences weekly. The construction contractor shall also keep logs of all activity performed under their direction, including records of corrective actions which were addressed.

SECTION D – DATA VALIDATION AND USABILITY

D1. Data Review, Verification, and Validation

Upon receipt of observation reports, inspection reports, and laboratory reports the QA/QC manager shall conduct a 100% completeness check to ensure all necessary information has been provided. Instances where information is missing shall be raised to the report-preparer for resolution. In the event that results are outside the expected range for the material, results will be flagged and the project team will investigate attributable causes. All resolutions will be documented.

D2. Verification and Validation Methods

Throughout the project, REL will perform bathymetric surveys to document incremental progress on the project that can be compared with Sevenson's reported progress. Additionally, volumes of material hauled to the landfill and Lot 24 can be used to verify estimated removal volumes. In isolated areas, discrepancies in bathymetry will be reconciled on a case by case basis.

D3. Reconciliation with User Requirements

In areas where underlying sediments are beneficially used, visual examination will be used to confirm that environmental spoils have been removed and chemical testing will confirm operational procedures based on assumptions in the conceptual site model. If visual assessment of sediment samples indicate presence of environmental spoils, additional dredging may be necessary. Re-dredging decisions may be based on a combination of visual examination and chemical testing. Once sampling confirms that environmental spoils have been removed acceptable sediment removal will be determined by achieving elevation specifications as determined by bathymetry.

In areas where the full sediment thickness is dredged and historic characterization indicates navigational spoils at the target elevation, acceptable performance will be determined by achieving target elevation specifications as determined by bathymetry.

In sediment removal areas where historic characterizations indicated contaminant concentrations near the TEC at depth, project objectives are met if surface sediments are below the following TECs:

9.8 mg/kg arsenic;
32 mg/kg copper;
36 mg/kg lead;
0.18 mg/kg mercury
120 mg/kg for zinc

As part of demobilization efforts, the on land staging area will dismantled in a manner that ensures that arsenic-contaminated materials are handled appropriately. The shoreline in the loading area is free of contaminated sediments, side slopes are stable and shoreline treatments or armoring are in place.

Habitat restoration area

If a combination of visual examination and chemical testing of sediment samples indicate potential presence of sediments with contaminant concentrations above the TEC, additional sampling may be necessary and operations for placing sediments in this area may be adjusted to better ensure clean sediments at the surface. Project objectives will be met when design elevations are achieved and concentrations of target metals on the surface do not exceed the TEC. In the event that any final surface sediment results exceed the TEC, WDNR and the City of Marinette will evaluate the sample concentrations and consider options including but not limited to whether material will need to be removed or additional clean material should be placed in the affected area.

Site Restoration and As Built Drawings

The technical specifications identify expectations for site closure and restoration as well as the expected conditions for Lot 24. REL, on behalf of the owner will assure contract requirements have been met in accordance with the specifications. Applicable specifications include but are not limited to:

31 81 92 sections 3.02 Site Condition and 3.03 Restoration of Disturbed Areas
35 20 13 sections 3.06 Post Construction Clean-up and 3.07 Final Examination and Acceptance

Once the project construction activities are completed, REL is responsible for providing As Built drawings for the harbor, staging areas and Lot 24.

APPENDICES

Appendix A – Plan Set

Appendix B – Sevenson Work & Dewatering Plan

Appendix C – Technical Specifications (abbreviated)

Appendix D – Wastewater Treatment System Monitoring Plan

Appendix E – Waste Management Agreement

Appendix F – Standard Operating Procedures (Sevenson and Robert E. Lee)

Appendix G – Quality Assurance Manual and Standard Operating Procedures (Pace Analytical)

Appendix H – Resumes for Sevenson & REL

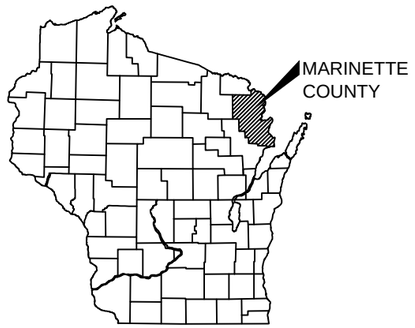
Appendix I – Sample Tables

APPENDIX A

Plan Set

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

JUNE 2014



VICINITY MAP
NTS

DRAWING INDEX	
SHT NO	DESCRIPTION
G1.0	TITLE SHEET
G1.1	LEGEND & ABBREVIATIONS
C1.0	ANTICIPATED DREDGE SPOILS TRUCK ROUTES
C2.0	LOT 24 EXISTING CONDITIONS - NAVIGATIONAL DREDGE SPOILS CONTAINMENT SITE
C2.1	LOT 24 LAYOUT - NAVIGATIONAL DREDGE SPOILS CONTAINMENT SITE
C2.2	LOT 24 BASE DESIGN - NAVIGATIONAL DREDGE SPOILS CONTAINMENT SITE
C2.3	LOT 24 FINAL GRADES - NAVIGATIONAL DREDGE SPOILS CONTAINMENT SITE
C2.4	CROSS SECTION
C3.0	EXISTING HARBOR SITE PLAN & CONTOURS
C3.1	HARBOR DREDGING PLAN & INTERMEDIARY CONTOURS
C3.2	FINAL HARBOR SITE PLAN & CONTOURS
C4.0	HARBOR CROSS SECTION - 1
C4.1	HARBOR CROSS SECTION - 2
C4.2	HARBOR CROSS SECTION - 3
C4.3	HARBOR CROSS SECTION - 4
C4.4	HARBOR CROSS SECTION - 5
C5.0	TYPICAL CROSS SECTIONS - 1
C5.1	TYPICAL CROSS SECTIONS - 2
C6.0	MISC. SITE DETAILS
S1.0	SHEET PILE WALL PLAN & PROFILE
S1.1	SHEET PILE WALL DETAILS
S2.0	STRUCTURAL DETAILS

NOTE:
THIS PLAN SET WAS PREPARED IN COLOR. REPRODUCTION BY MEANS OTHER THAN EQUIVALENT COLOR COPYING MAY CAUSE SOME DATA TO BE LOST OR MISREPRESENTED.



PROJECT AREA

NORTH

DES BY G. SHAMBEAU	BOOK NO ----							MENEKAUNEE HARBOR IMPROVEMENTS CITY OF MARINETTE MARINETTE, WISCONSIN	AYRES ASSOCIATES EAU CLAIRE, WISCONSIN	TITLE SHEET	SHEET NO G1.0
DR BY G. SHAMBEAU	PROJ NO 19-0277.33										
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION				

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EXISTING LEGEND:

SANITARY SEWER

WATER MAIN

STORM SEWER

UNDERGROUND GAS

UNDERGROUND ELECTRIC

UNDERGROUND FIBER OPTIC

UNDERGROUND TELEPHONE

UNDERGROUND VIDEO

OVERHEAD ELECTRIC

UTILITY POLES: POWER POLES

TELEPHONE POLE

LIGHT POLE

CONTROL POINT

PEDESTAL / TRANSFORMER

CURB AND GUTTER

SIDEWALK

HARD SURFACED ROADWAY

NON-SURFACED ROADWAY

TREE - DECIDUOUS

TREE - CONIFEROUS

TREE LINE

BUSH

CONTOURS

DITCH

FENCE

RIGHT OF WAY

PROPERTY LINE

SECTION LINE

RETAINING WALL

WETLAND

STREET CENTERLINE

WATER'S EDGE

SWAMP EDGE

BENCHMARK

BUILDING

PROPOSED LEGEND:

SANITARY SEWER

FORCE MAIN

WATER MAIN

STORM SEWER

CURB AND GUTTER

SIDEWALK

HARD SURFACE ROADWAY

NON-SURFACED ROADWAY

CONTOURS

FENCE

BASELINE

PERMANENT EASEMENT

TEMPORARY EASEMENT

SILT FENCE

EROSION BALES

EROSION MAT

EROSION LOG

SAW CUT

DITCH

RETAINING WALL

POND

SPOT ELEVATION

CONSTRUCTION EASEMENT

UTILITY EASEMENT

SETBACK

GUARD RAIL

ABBREVIATIONS:

AB	ANCHOR BOLT
ABV	ABOVE
ADJ	ADJUST
AFF	ABOVE FINISHED FLOOR
AL	ALUMINUM
ALT	ALTERNATE WITH
ALT/	ALTERNATE
APPROX	APPROXIMATE
ASPH	ASPHALT
AUTO	AUTOMATIC
AVE	AVENUE
@	AT
B CRS	BASE COURSE
BC	BACK OF CURB
BD	BOARD
BEL	BELOW
BETWN	BETWEEN
BF	BACK FACE
BFV	BUTTERFLY VALVE
BIT	BITUMINOUS
BLD	BLIND
BLDG	BUILDING
BLK	BLOCK
BLKG	BLOCKING
BLVD	BOULEVARD
BM	BENCHMARK
BO	BREAKOFF
BRG	BEARING
BRK	BRICK
BS	BACK OF SIDEWALK
BTM	BOTTOM
BV	BALL VALVE
℄	BASELINE
B/	BOTTOM OF
C&G	CURB AND GUTTER
CB	CATCH BASIN
CF	CUBIC FOOT
CHKD P	CHECKERED PLATE
CI	CAST IRON
CJ	CONTROL JOINT
CL / ℄	CENTERLINE
CHL	CHLORINE
CLG	CEILING
CLR	CLEAR
CMP	CORRUGATED METAL PIPE
CMU	CONCRETE MASONRY UNIT
CO	CLEANOUT
CONC	CONCRETE
CONN	CONNECTION
CONST	CONSTRUCTION
CONST JT	CONSTRUCTION JOINT
CONT	CONTINUOUS
CONTR	CONTRACTOR
CONTR JT	CONTRACTION JOINT
COR	CORNER
CP	CONTROL POINT
CPLG	COUPLING
CRS	COURSE
CSP	CORRUGATED STEEL PIPE
CTG	CASTING
CTH	COUNTY TRUNK HIGHWAY
CULV	CULVERT
CV	CHECK VALVE
CW	COLD WATER
CY	CUBIC YARD
DEFL	DEFLECTION
DEG / °	DEGREE
DF	DRINKING FOUNTAIN
DI	DUCTILE IRON
DIA	DIAMETER
DIM	DIMENSION
DISCH	DISCHARGE
DN	DOWN
DP	DEPTH
DR	DOOR
DTL	DETAIL
DW	DRIVEWAY
DWG	DRAWING
E	EAST
EA	EACH
EF	EACH FACE
EFF	EFFLUENT
EJ	EXPANSION JOINT
EL	ELEVATION
ELB	ELBOW
ELEC	ELECTRICAL
EQ	EQUAL
EQUIP	EQUIPMENT
ER	END OF RADIUS
EST	ESTIMATE
EW	EACH WAY
EWC	ELECTRIC WATER COOLER
EXG	EXISTING
EXH	EXHAUST
EXP	EXPANSION
EXT	EXTERIOR
FD	FLOOR DRAIN
FDN	FOUNDATION
FERT	FERTILIZER
FF	FAR FACE
FGL	FIBERGLASS
FIN	FINISHED
FL	FLOWLINE
FLG	FLANGED
FLR	FLOOR
FM	FORCE MAIN
FN	FENCE
FT (')	FOOT
FTG	FOOTING
FUT	FUTURE
G	GAS
GA	GAGE
GAR	GARAGE
GEN	GENERAL
GRD	GRADE, GROUND
GV	GATE VALVE
GRAV	GRAVEL
GW	GROUNDWATER
HB	HOSE BIB
HM	HOLLOW METAL
HORZ	HORIZONTAL
HSE	HOUSE
HT	HEIGHT
HW	HOT WATER
HWR	HOT WATER RETURN
HYD	HYDRANT
ID	INSIDE DIAMETER
IF	INSIDE FACE
IN (")	INCHES
INCL	INCLUDE
INF	INFLUENT
INL	INLET
INSUL	INSULATION
INT	INTERIOR
INV	INVERT
IP	IRON PIPE
IPS	IRON PIPE SIZE
JT	JOINT
KGV	KNIFE GATE VALVE
LAB	LABORATORY
LAV	LAVATORY
LC	LENGTH OF CURVE
LF	LINEAL FEET
LG	LENGTH
LOC	LOCATION
LP	LIGHT POLE
LR	LONG RADIUS
LS	LUMP SUM
LT	LEFT
MAS	MASONRY
MAX	MAXIMUM
MECH	MECHANICAL
MFG	MANUFACTURER
MH	MANHOLE
MIN	MINIMUM
MISC	MISCELLANEOUS
MJ	MECHANICAL JOINT
MP	MID POINT
MTL	MATERIAL
N	NORTH
NF	NEAR FACE
NIC	NOT IN CONTRACT
NO	NUMBER
NOM	NOMINAL
NPW	NON-POTABLE WATER
NTS	NOT TO SCALE
OC	ON CENTER
OD	OUTSIDE DIAMETER
OE	OVERHEAD ELECTRIC
OF	OUTSIDE FACE
OH	OVERHEAD
OPG	OPENING
OT	OVERHEAD TELEPHONE
PC	POINT OF CURVE
PE	PRIVATE ENTRANCE
PED	PEDESTAL
PERF	PERFORATE
PI	POINT OF INTERSECTION
PKG	PARKING
P	PLATE
PL	PLACE
PL ℄	PROPERTY LINE
PP	POWER POLE
PSF	POUNDS PER SQUARE FOOT
PSI	POUNDS PER SQUARE INCH
PT	POINT OF TANGENCY
PV	PLUG VALVE
PVC	POLYVINYL CHLORIDE
PVMT	PAVEMENT
PW	POTABLE WATER
%	PERCENT
QTY	QUANTITY
RAD	RADIUS
RCP	REINFORCED CONCRETE PIPE
RD	ROAD
RD	ROOF DRAIN
RDWY	ROADWAY
RED	REDUCER
REF	REFERENCE
REINF	REINFORCING
REM	REMOVE
REPL	REPLACE
REQD	REQUIRED
REV	REVISED
RM	ROOM
RR	RAILROAD
RT	RIGHT
RW	RIGHT OF WAY
S	SOUTH
SAN	SANITARY
SAMH	SANITARY MANHOLE
SCH	SCHEDULE
SEC	SECTION
SF	SQUARE FOOT
SHT	SHEET
SIM	SIMILAR
SL	SLOPE
SPA	SPACE
SPEC	SPECIFICATION
SQ	SQUARE
SS	STAINLESS STEEL
SSMH	STORM SEWER MANHOLE
ST	STREET
STD	STANDARD
STH	STATE TRUNK HIGHWAY
STL	STEEL
STM	STORM
STP	SEWAGE TREATMENT PLANT
SURF	SURFACE
SW	SIDEWALK
SWR	SEWER
SY	SQUARE YARD
SYS	SYSTEM
T&B	TOP & BOTTOM
TC	TOP OF CURB
TELE	TELEPHONE
THK	THICKNESS
THRU	THROUGH
TP	TELEPHONE POLE
TYP	TYPICAL
T/	TOP OF
UG	UNDERGROUND GAS
UE	UNDERGROUND ELECTRICAL
UNEXC	UNEXCAVATED
UNO	UNLESS NOTED OTHERWISE
USH	UNITED STATES HIGHWAY
UT	UNDERGROUND TELEPHONE
UV	UNDERGROUND VIDEO
V	VALVE
V&B	VALVE & BOX
VAR	VARIABLE
VER	VERTICAL
VC	VERTICAL CURVE
W	WEST
W/	WITH
W/O	WITHOUT
WD	WIDTH
WM	WATER MAIN
WS	WATER SURFACE
WTP	WATER TREATMENT PLANT
WTR	WATER
WWF	WELDED WIRE FABRIC
WWM	WOVEN WIRE MESH
WWTP	WASTEWATER TREATMENT PLANT

LEGEND:

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00 / PR-12 — DETAIL OR PLAN NUMBER; SHEET WHERE DRAWN

NUMBER OF DRAWING ON THAT SHEET

1 PLAN, DETAIL OR SECTION
1" = 100'

DES BY	G. SHAMBEAU	BOOK NO	----	NO	DATE	REVISION	NO	DATE	REVISION	MENEKAUNEE HARBOR IMPROVEMENTS CITY OF MARINETTE MARINETTE, WISCONSIN	AYRES ASSOCIATES EAU CLAIRE, WISCONSIN	LEGEND & ABBREVIATIONS	SHEET NO G1.1
DR BY	G. SHAMBEAU	PROJ NO	19-0277.33										
CHK BY	C. GOODWIN	DATE	JUNE 2014										

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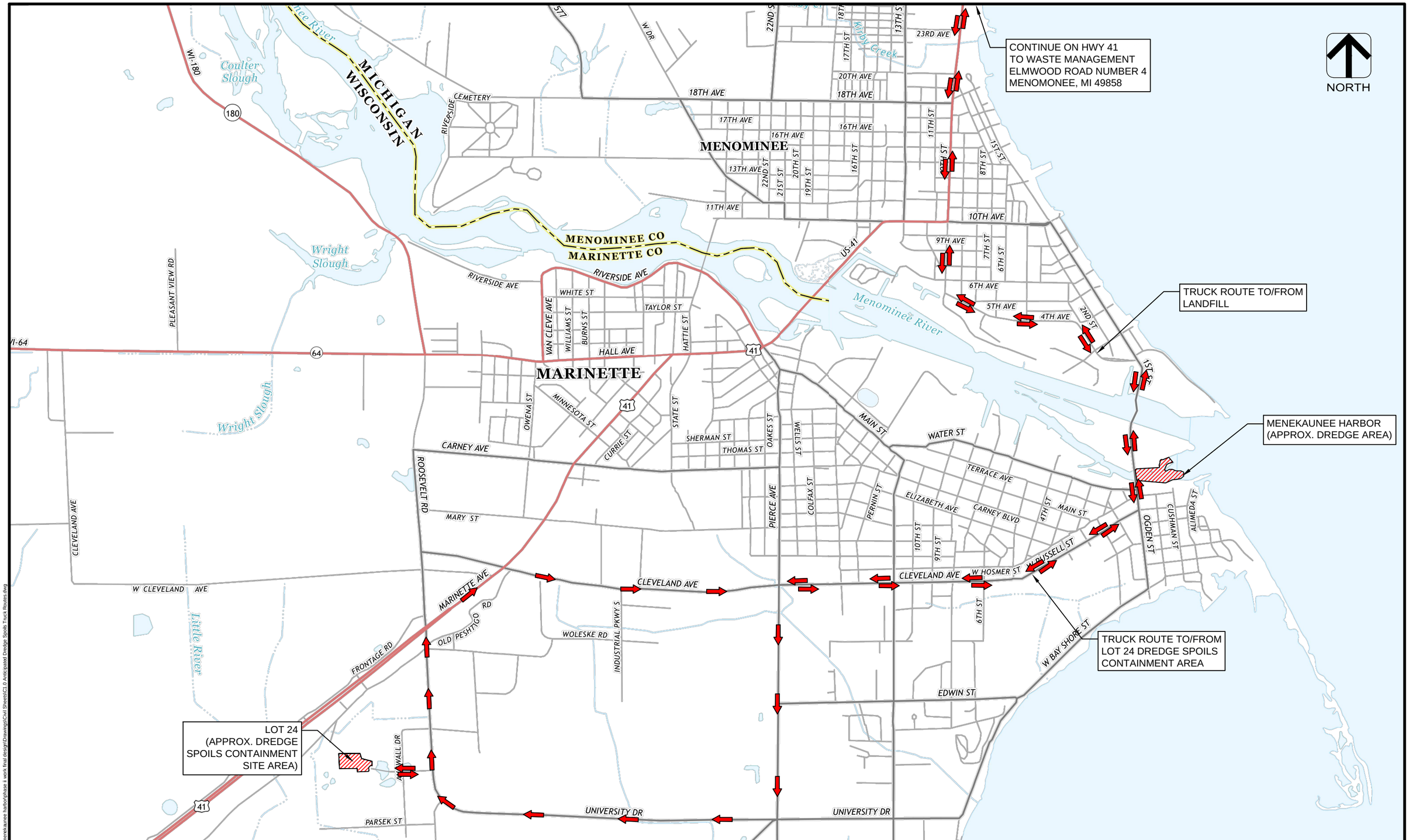

DES BY	G. SHAMBEAU	BOOK NO	----
DR BY	G. SHAMBEAU	PROJ NO	19-0277.33
CHK BY	C. GOODWIN	DATE	JUNE 2014

NO	DATE	REVISION	NO	DATE	REVISION

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
EAU CLAIRE, WISCONSIN

ANTICIPATED DREDGE SPOILS TRUCK ROUTES

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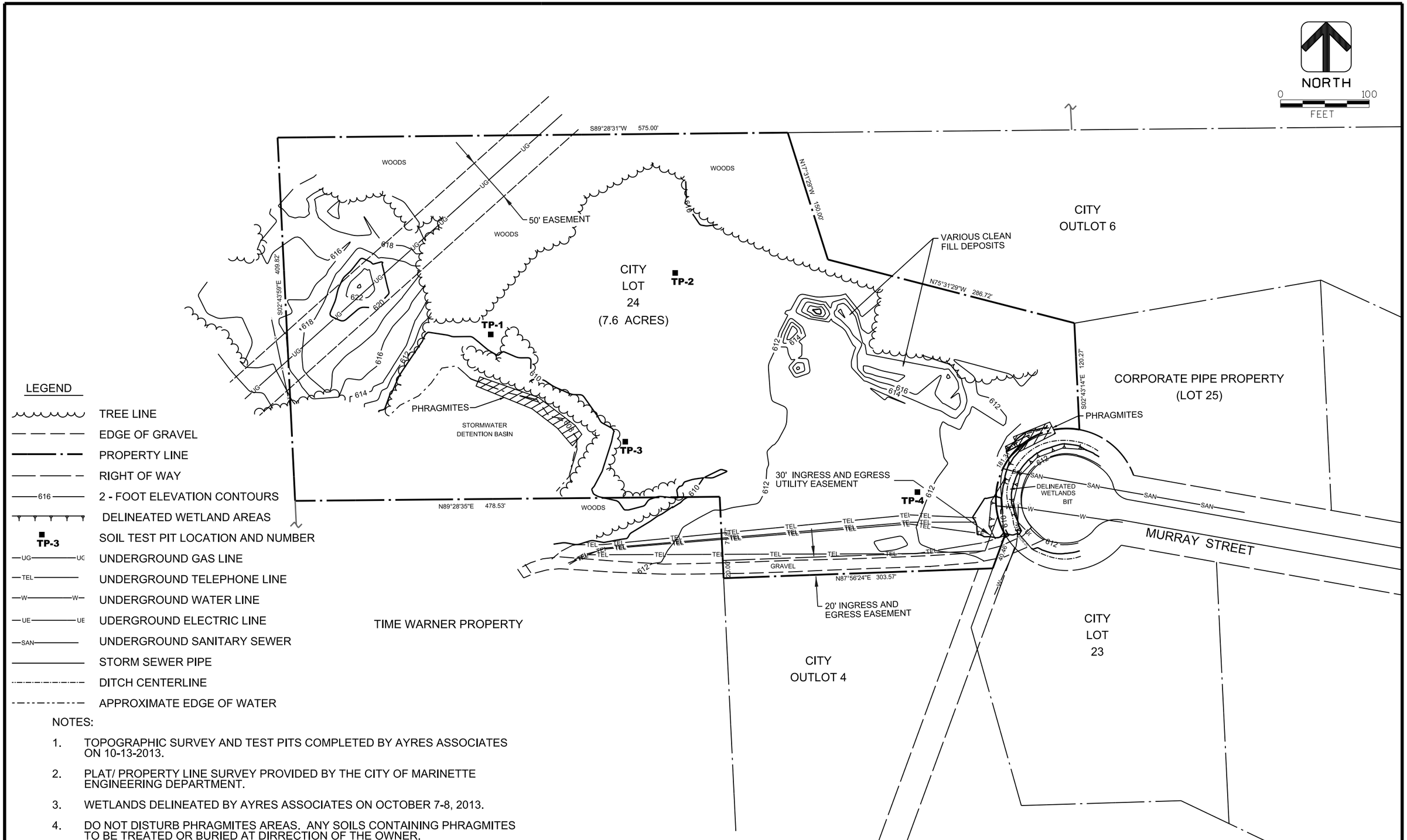

NOTES:

1. TOPOGRAPHIC SURVEY AND TEST PITS COMPLETED BY AYRES ASSOCIATES ON 10-13-2013.
2. PLAT/ PROPERTY LINE SURVEY PROVIDED BY THE CITY OF MARINETTE ENGINEERING DEPARTMENT.
3. WETLANDS DELINEATED BY AYRES ASSOCIATES ON OCTOBER 7-8, 2013.
4. DO NOT DISTURB PHRAGMITES AREAS. ANY SOILS CONTAINING PHRAGMITES TO BE TREATED OR BURIED AT DIRRECTION OF THE OWNER.

DR.BY T. SHUPERT	BOOK NO.				3	March 19, 2014	100% Design Level Plans
CHK.BY D. FREE	JOB NO. 19-0277.36				2	Feb. 18, 2014	60% Design Level Plans
DATE JUNE 2014	SCALE AS SHOWN	NO.	DATE	REVISION	1	Nov. 15, 2013	30% Design Level Plans
					NO.	DATE	REVISION

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
Eau Claire, Wisconsin

LOT 24 EXISTING CONDITIONS
NAVIGATIONAL DREDGE SPOILS CONTAINMENT SITE

DRAWING NO. C2.0

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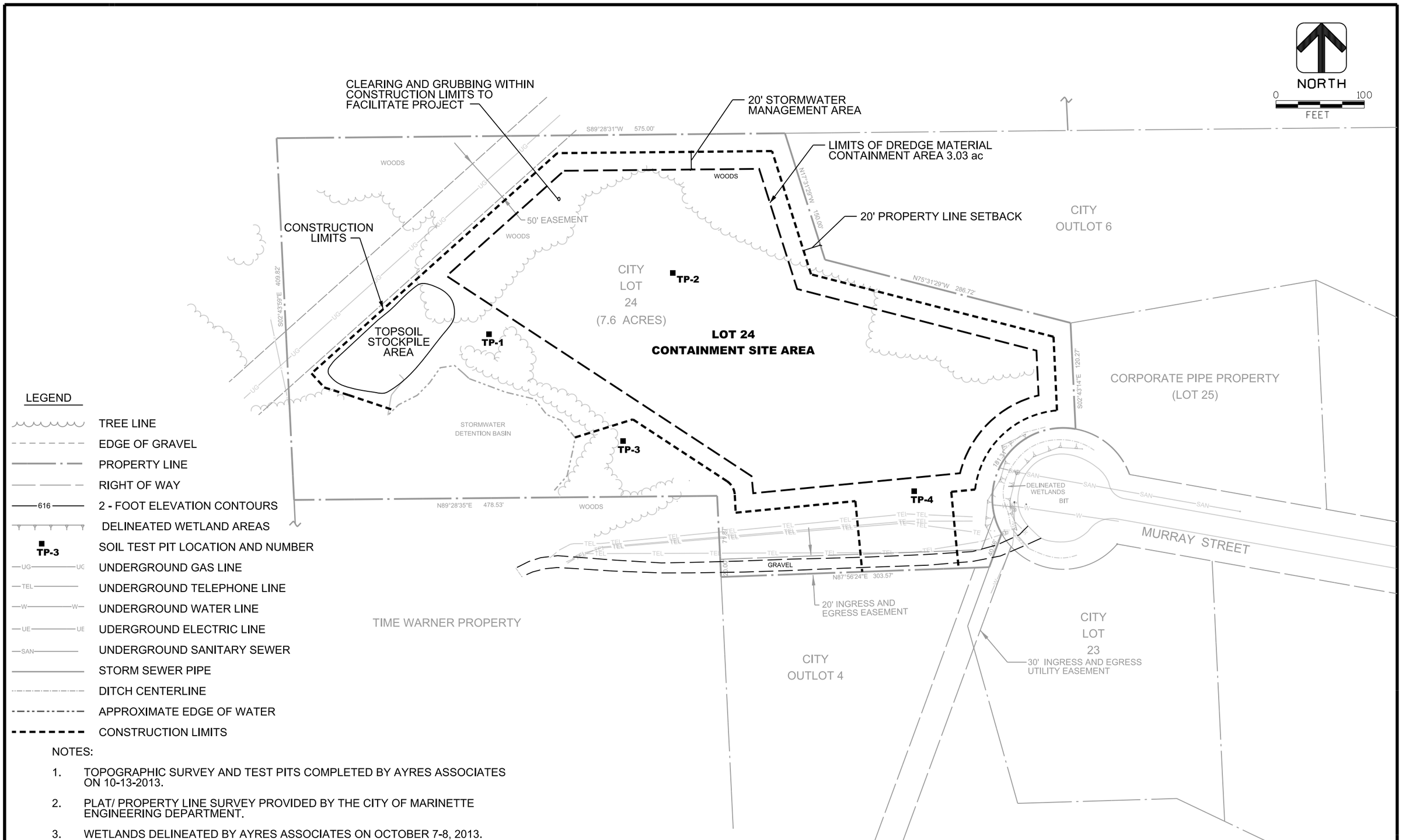

LEGEND

- TREE LINE
- EDGE OF GRAVEL
- PROPERTY LINE
- RIGHT OF WAY
- 616 — 2 - FOOT ELEVATION CONTOURS
- DELINEATED WETLAND AREAS
- TP-3 — SOIL TEST PIT LOCATION AND NUMBER
- UG — UG — UNDERGROUND GAS LINE
- TEL — UNDERGROUND TELEPHONE LINE
- W — W — UNDERGROUND WATER LINE
- UE — UE — UDERGROUND ELECTRIC LINE
- SAN — UNDERGROUND SANITARY SEWER
- STORM SEWER PIPE
- DITCH CENTERLINE
- APPROXIMATE EDGE OF WATER
- CONSTRUCTION LIMITS

NOTES:

1. TOPOGRAPHIC SURVEY AND TEST PITS COMPLETED BY AYRES ASSOCIATES ON 10-13-2013.
2. PLAT/ PROPERTY LINE SURVEY PROVIDED BY THE CITY OF MARINETTE ENGINEERING DEPARTMENT.
3. WETLANDS DELINEATED BY AYRES ASSOCIATES ON OCTOBER 7-8, 2013.

DR,BY	T. SHUPERT
CHK,BY	D. FREE
DATE	JUNE 2014
BOOK NO.	
JOB NO.	19-0277.36
SCALE	AS SHOWN

NO.	DATE	REVISION

NO.	DATE	REVISION
3	March 19, 2014	100% Design Level Plans
2	Feb. 18, 2014	60% Design Level Plans
1	Nov. 15, 2013	30% Design Level Plans

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
Eau Claire, Wisconsin

LOT 24 LAYOUT
NAVIGATIONAL DREDGE SPOILS CONTAINMENT SITE

DRAWING NO.
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EARTHWORK ESTIMATES
TOPSOIL AVE EXCAVATION DEPTH = 6 IN.
TOTAL TOSOIL EXCAVATION = 2,440 CY

NORTH
0 100
FEET

SILT FENCE SITE PERIMETER
20' STORMWATER MANAGEMENT AREA
LIMITS OF DREDGE MATERIAL CONTAINMENT AREA 3.03 ACRES
20' PROPERTY LINE SETBACK
6" AVERAGE DEPTH OF TOPSOIL TO BE STRIPPED AND STOCKPILED. SLOPE AREA TO PERIMETER SWALE AT SOUTH ONLY AND TOWARD DRAINGAGEWAY
PLACE 3" TO 4" OF SHREDDED WOOD ON TOP OF PREPARED BASE PRIOR TO PLACEMENT OF SPOILS
SHREDDED WOOD PLACED PERIODICALLY TO SUPPORT TRAFFIC INTO AND OUT OF CONTAINMENT SITE AREA
NO PROJECT DRAINAGE ALLOWED OFF-SITE OR TO WETLAND
CONSTRUCTION LIMITS
TOPSOIL STOCKPILE AREA
SLOPE BASE OF CONTAINMENT AREA TOWARD DRAINAGEWAY
8' WIDE STONE DRAINAGEWAY
BASE DRAINAGE (TYPICAL)
INSTALL SITE ACCESS ROAD
PERIMETER DRAINAGE SWALE TO SLOPE AT MINIMUM OF 0.10% AROUND PERIMETER TOWARD DRAINAGEWAY AND INTO DETENTION BASIN
12" CMP CULVERT UNDER ROAD ACCESS
20' INGRESS AND EGRESS EASEMENT
30' INGRESS AND EGRESS
~0.1%
S89°28'31"W 575.00'
N17°31'29"W 150.00'
N75°31'29"W 286.72'
S02°43'59"E 409.82'
S02°43'14"E 120.27'
N89°28'35"E 478.53'
N87°56'24"E 303.57'
50' EASEMENT
WOODS
CITY LOT 24
STORMWATER DETENTION BASIN
CITY OUTLOT 6
CORPORATE PIPE PROPERTY (LOT 25)
DELINEATED WETLANDS
BIT
MURRAY STREET
GRAVEL
TIME WARNER PROPERTY
CITY OUTLOT 4
CITY LOT 23

LEGEND

- TREE LINE
- EDGE OF GRAVEL
- PROPERTY LINE
- RIGHT OF WAY
- 616 — 2 - FOOT ELEVATION CONTOURS
- DELINEATED WETLAND AREAS
- UG — UNDERGROUND GAS LINE
- TEL — UNDERGROUND TELEPHONE LINE
- W — UNDERGROUND WATER LINE
- UE — UDERGROUND ELECTRIC LINE
- SAN — UNDERGROUND SANITARY SEWER
- STORM SEWER PIPE
- DITCH CENTERLINE
- APPROXIMATE EDGE OF WATER
- CONSTRUCTION LIMITS
- SF — SILT FENCE

NOTES:

1. TOPOGRAPHIC SURVEY AND TEST PITS COMPLETED BY AYRES ASSOCIATES ON 10-13-2013.
2. PLAT/ PROPERTY LINE SURVEY PROVIDED BY THE CITY OF MARINETTE ENGINEERING DEPARTMENT.
3. WETLANDS DELINEATED BY AYRES ASSOCIATES ON OCTOBER 7-8, 2013.

DR,BY	T. SHUPERT
CHK,BY	D. FREE
DATE	JUNE 2014
BOOK NO.	
JOB NO.	19-0277.36
SCALE	AS SHOWN

NO.	DATE	REVISION

NO.	DATE	REVISION
3	March 19, 2014	100% Design Level Plans
2	Feb. 18, 2014	60% Design Level Plans
1	Nov. 15, 2013	30% Design Level Plans

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
Eau Claire, Wisconsin

LOT 24 BASE DESIGN
NAVIGATIONAL DREDGE SPOILS CONTAINMENT SITE

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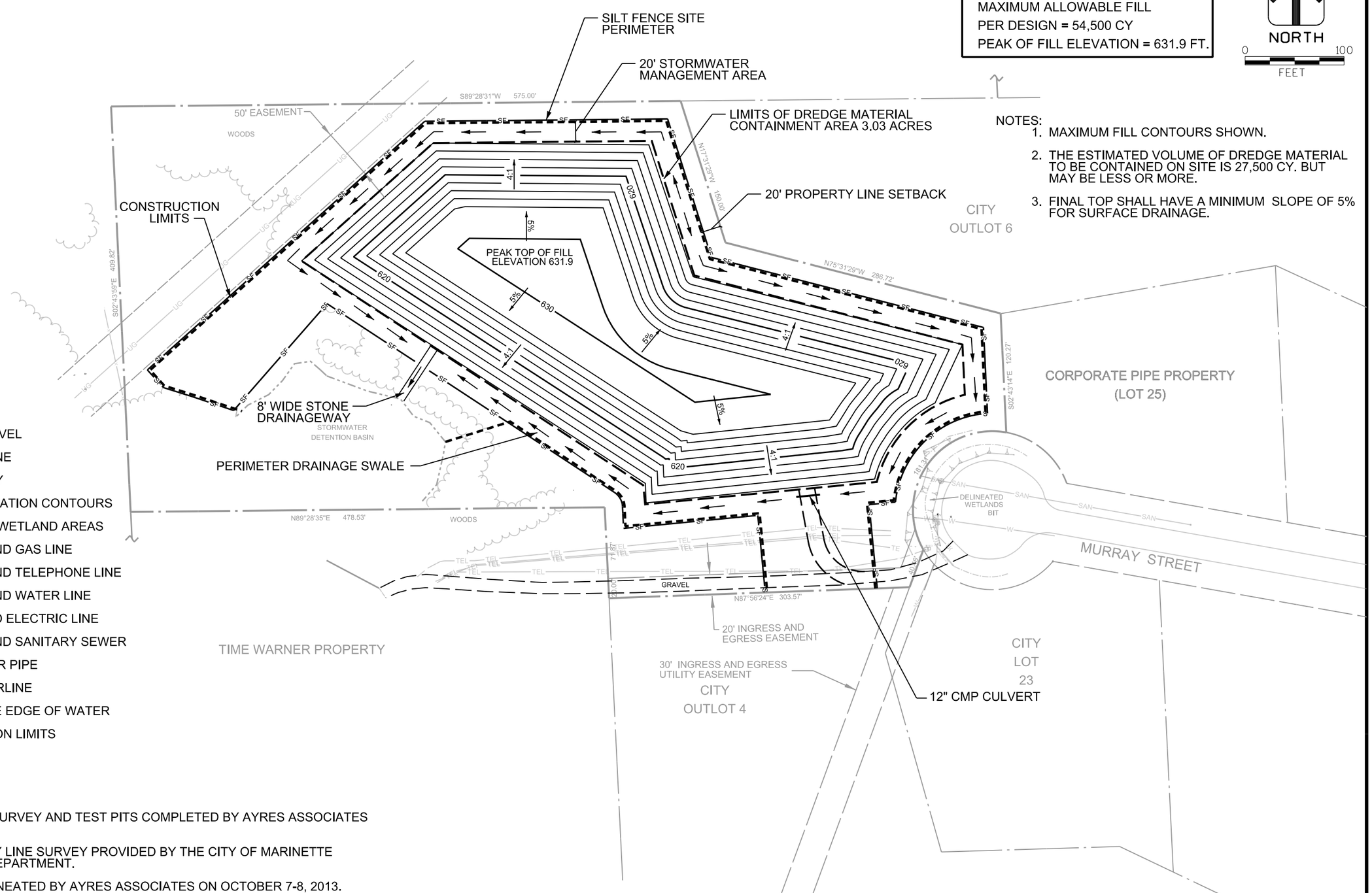
EARTHWORK ESTIMATES
MAXIMUM ALLOWABLE FILL
PER DESIGN = 54,500 CY
PEAK OF FILL ELEVATION = 631.9 FT.

NORTH
0 100
FEET

NOTES:
1. MAXIMUM FILL CONTOURS SHOWN.
2. THE ESTIMATED VOLUME OF DREDGE MATERIAL TO BE CONTAINED ON SITE IS 27,500 CY. BUT MAY BE LESS OR MORE.
3. FINAL TOP SHALL HAVE A MINIMUM SLOPE OF 5% FOR SURFACE DRAINAGE.



LEGEND

- TREE LINE
- EDGE OF GRAVEL
- PROPERTY LINE
- RIGHT OF WAY
- 616 — 2 - FOOT ELEVATION CONTOURS
- DELINEATED WETLAND AREAS
- UG — UNDERGROUND GAS LINE
- TEL — UNDERGROUND TELEPHONE LINE
- W — UNDERGROUND WATER LINE
- UE — UDERGROUND ELECTRIC LINE
- SAN — UNDERGROUND SANITARY SEWER
- STORM SEWER PIPE
- DITCH CENTERLINE
- APPROXIMATE EDGE OF WATER
- CONSTRUCTION LIMITS
- SF — SILT FENCE

NOTES:
1. TOPOGRAPHIC SURVEY AND TEST PITS COMPLETED BY AYRES ASSOCIATES ON 10-13-2013.
2. PLAT/ PROPERTY LINE SURVEY PROVIDED BY THE CITY OF MARINETTE ENGINEERING DEPARTMENT.
3. WETLANDS DELINEATED BY AYRES ASSOCIATES ON OCTOBER 7-8, 2013.

DR.BY T. SHUPERT	BOOK NO.			3	March 19, 2014	100% Design Level Plans
CHK.BY D. FREE	JOB NO. 19-0277.36			2	Feb. 18, 2014	60% Design Level Plans
DATE JUNE 2014	SCALE AS SHOWN	NO.	DATE	REVISION	1	Nov. 15, 2013	30% Design Level Plans

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
Eau Claire, Wisconsin

LOT 24 FINAL GRADES
NAVIGATIONAL DREDGE SPOILS CONTAINMENT SITE

DRAWING NO. C2.3

6/5/2014
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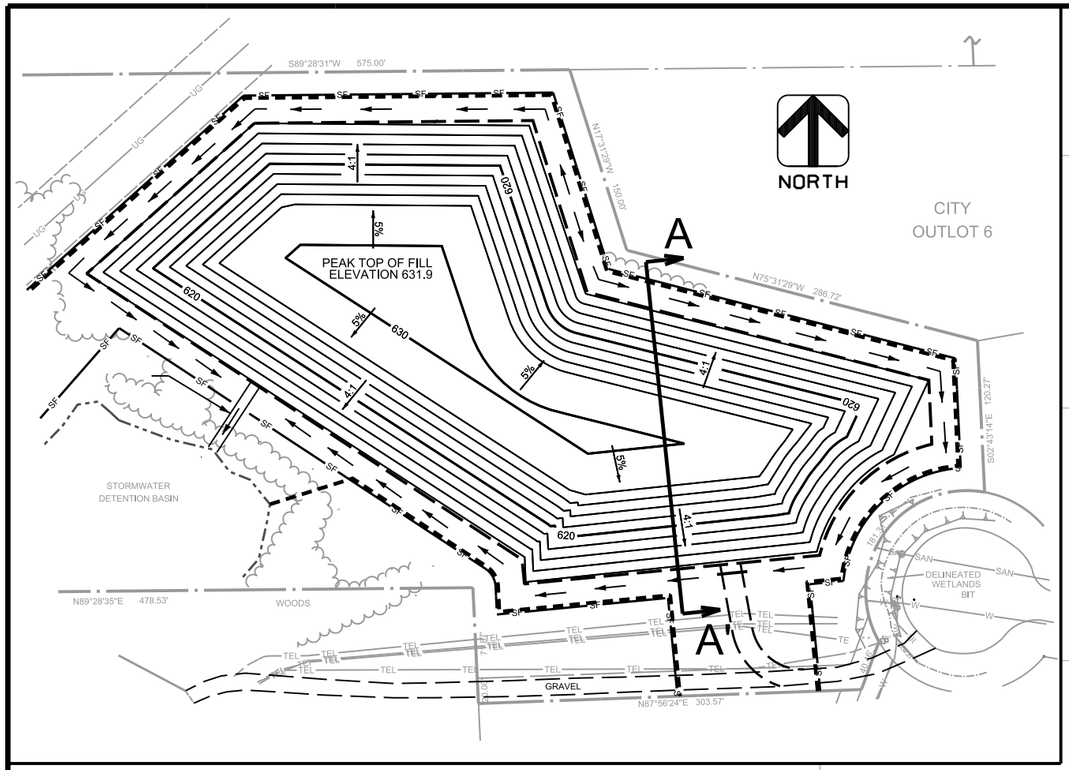
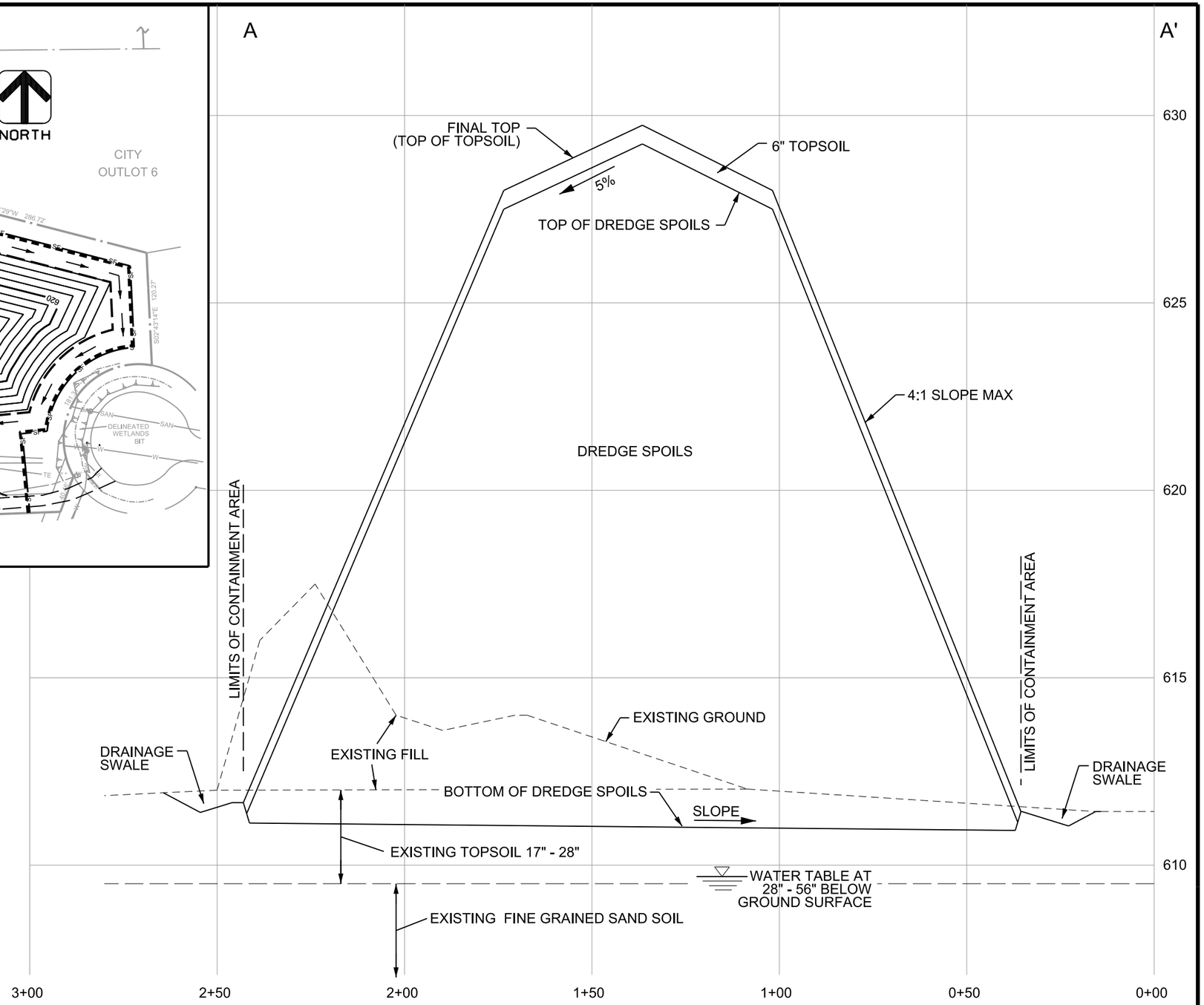
V:\ENV\CAD\LANDFILL\Mekaunee Harbor lot 24 fill\Lot 24 Top of Fill.dgn



SECTION LOCATION

NOT TO SCALE



CROSS SECTION A - A'

LOOKING EAST

6/5/2014
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W:\Bentley_V8i\WorkSpace\Projects\Engineering Standards.cfg\ES_Standard.tbl
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DR,BY	BOOK NO.
T. SHUPERT	
CHK,BY	JOB NO.
D. FREE	19-0277.36
DATE	SCALE
JUNE 2014	AS SHOWN

NO.	DATE	REVISION

NO.	DATE	REVISION
3	March 19, 2014	100% Design Level Plans
2	Feb. 18, 2014	60% Design Level Plans
1	Nov. 15, 2013	30% Design Level Plans

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

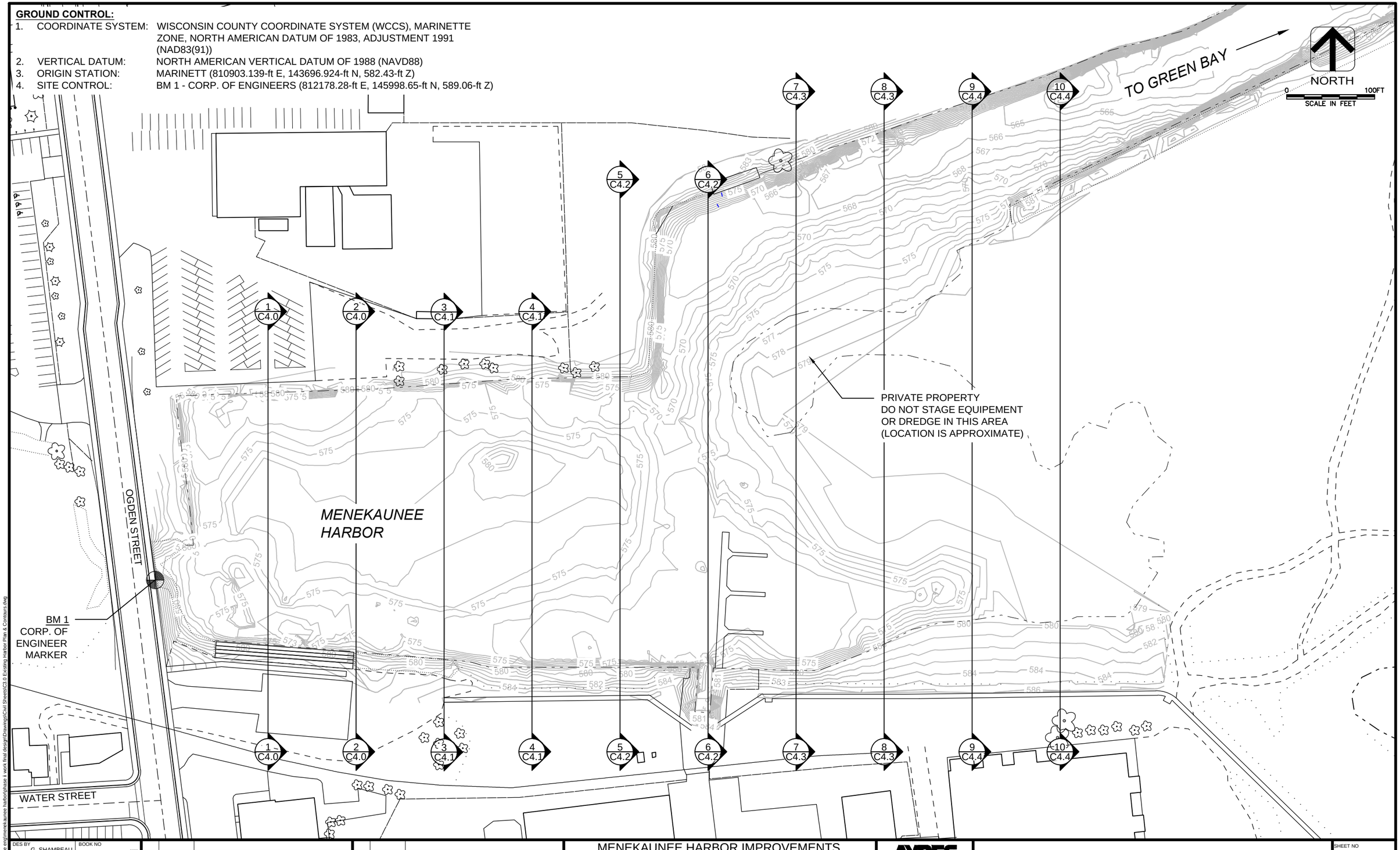
AYRES ASSOCIATES
Eau Claire, Wisconsin

CROSS SECTION

DRAWING NO.
C2.4

GROUND CONTROL:

1. COORDINATE SYSTEM: WISCONSIN COUNTY COORDINATE SYSTEM (WCCS), MARINETTE ZONE, NORTH AMERICAN DATUM OF 1983, ADJUSTMENT 1991 (NAD83(91))
2. VERTICAL DATUM: NORTH AMERICAN VERTICAL DATUM OF 1988 (NAVD88)
3. ORIGIN STATION: MARINETT (810903.139-ft E, 143696.924-ft N, 582.43-ft Z)
4. SITE CONTROL: BM 1 - CORP. OF ENGINEERS (812178.28-ft E, 145998.65-ft N, 589.06-ft Z)



DES BY G. SHAMBEAU	BOOK NO ----						MENEKAUNEE HARBOR IMPROVEMENTS CITY OF MARINETTE MARINETTE, WISCONSIN	AYRES ASSOCIATES EAU CLAIRE, WISCONSIN	EXISTING HARBOR PLAN & CONTOURS	SHEET NO C3.0
DR BY G. SHAMBEAU	PROJ NO 19-0277.33									
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION			

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6/5/2014
k:\water resource eng\menekaunee harbor\phase ii work final design\Drawings\Civil Sheets\C3.0 Existing Harbor Plan & Contours.dwg

NOTES:

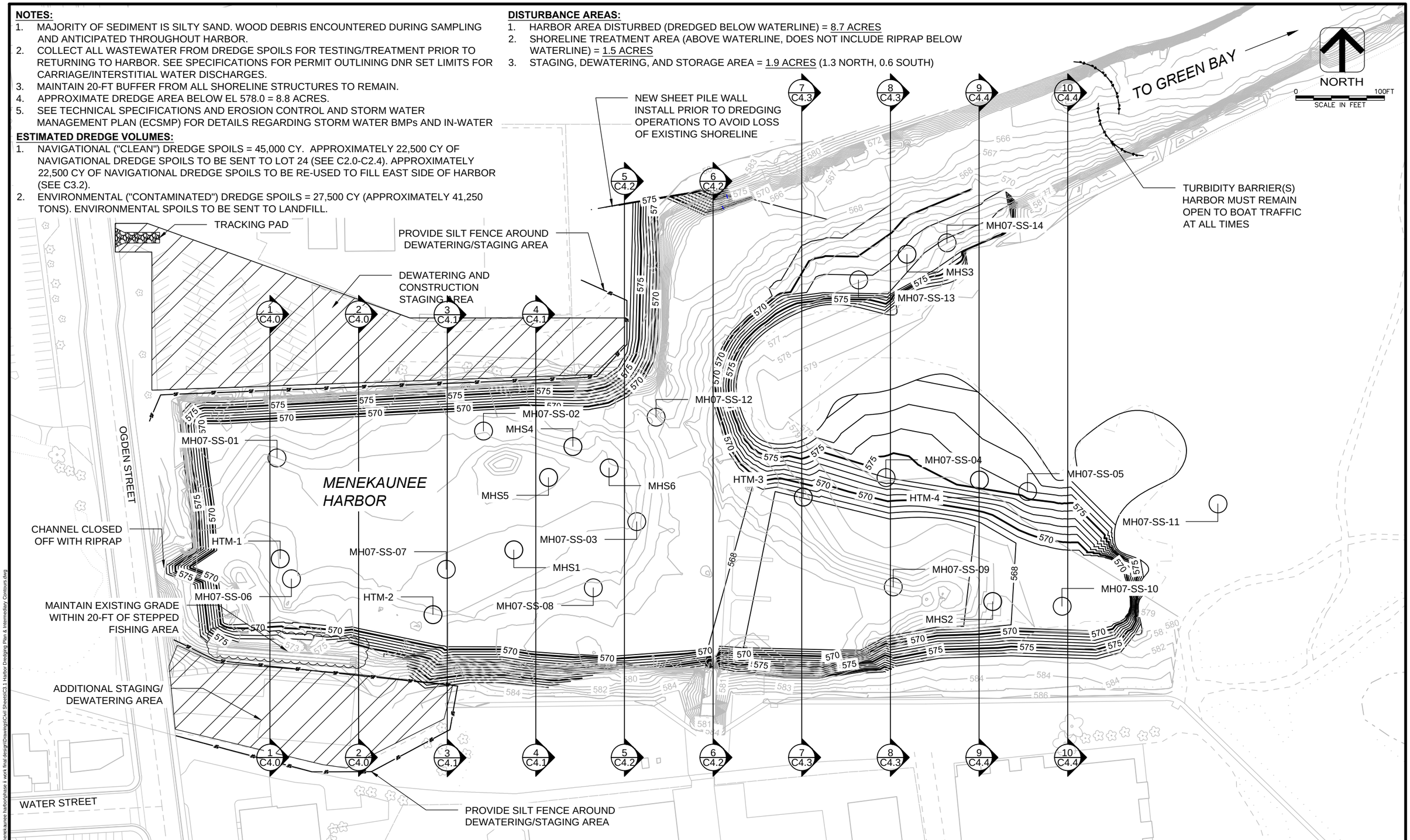
1. MAJORITY OF SEDIMENT IS SILTY SAND. WOOD DEBRIS ENCOUNTERED DURING SAMPLING AND ANTICIPATED THROUGHOUT HARBOR.
2. COLLECT ALL WASTEWATER FROM DREDGE SPOILS FOR TESTING/TREATMENT PRIOR TO RETURNING TO HARBOR. SEE SPECIFICATIONS FOR PERMIT OUTLINING DNR SET LIMITS FOR CARRIAGE/INTERSTITIAL WATER DISCHARGES.
3. MAINTAIN 20-FT BUFFER FROM ALL SHORELINE STRUCTURES TO REMAIN.
4. APPROXIMATE DREDGE AREA BELOW EL 578.0 = 8.8 ACRES.
5. SEE TECHNICAL SPECIFICATIONS AND EROSION CONTROL AND STORM WATER MANAGEMENT PLAN (ECSMP) FOR DETAILS REGARDING STORM WATER BMPs AND IN-WATER

ESTIMATED DREDGE VOLUMES:

1. NAVIGATIONAL ("CLEAN") DREDGE SPOILS = 45,000 CY. APPROXIMATELY 22,500 CY OF NAVIGATIONAL DREDGE SPOILS TO BE SENT TO LOT 24 (SEE C2.0-C2.4). APPROXIMATELY 22,500 CY OF NAVIGATIONAL DREDGE SPOILS TO BE RE-USED TO FILL EAST SIDE OF HARBOR (SEE C3.2).
2. ENVIRONMENTAL ("CONTAMINATED") DREDGE SPOILS = 27,500 CY (APPROXIMATELY 41,250 TONS). ENVIRONMENTAL SPOILS TO BE SENT TO LANDFILL.

DISTURBANCE AREAS:

1. HARBOR AREA DISTURBED (DREDGED BELOW WATERLINE) = 8.7 ACRES
2. SHORELINE TREATMENT AREA (ABOVE WATERLINE, DOES NOT INCLUDE RIPRAP BELOW WATERLINE) = 1.5 ACRES
3. STAGING, DEWATERING, AND STORAGE AREA = 1.9 ACRES (1.3 NORTH, 0.6 SOUTH)



DES BY	G. SHAMBEAU	BOOK NO	----
DR BY	G. SHAMBEAU	PROJ NO	19-0277.33
CHK BY	C. GOODWIN	DATE	JUNE 2014

NO	DATE	REVISION	NO	DATE	REVISION

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
EAU CLAIRE, WISCONSIN

HARBOR DREDGING PLAN & INTERMEDIARY CONTOURS

SHEET NO C3.1

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NEW SHEETPILE RETAINING WALL AND BOAT STAGING AREA (SEE DTL. 2 SHT. C5.1)

RIPRAP SHORELINE (SEE DTL. 1 SHT. C5.0)

RIPRAP SHORELINE (SEE DTL. 3 SHT. C5.0)

RIPRAP SHORELINE (SEE DTL. 1 SHT. C5.0)

WALKWAY TERMINATES AT HARBOR INLET (FUTURE BRIDGE WALK)

RIPRAP SHORELINE (SEE DTL. 2 SHT. C5.0)

FLAT GRADED AREA AT ELEVATION 583.0 FOR FUTURE STRUCTURE

RIPRAP SHORELINE (SEE DTL. 1 SHT. C5.0)

LEDGE STONE SHORELINE TREATMENT (SEE DTL. 1 SHT. S5.1)

WATER'S EDGE (EL. 578.0 ±)

AREA TO BE BACKFILLED WITH BENEFICIAL USE FILL

MENEKAUNEE HARBOR

OGDEN STREET

WATER STREET

TO GREEN BAY

NORTH

0 100FT

SCALE IN FEET

1 C4.0 | 2 C4.0 | 3 C4.1 | 4 C4.1 | 5 C4.2 | 6 C4.2 | 7 C4.3 | 8 C4.3 | 9 C4.4 | 10 C4.4

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DES BY G. SHAMBEAU	BOOK NO ----								
DR BY G. SHAMBEAU	PROJ NO 19-0277.33								
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION		

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
EAU CLAIRE, WISCONSIN

FINAL HARBOR PLAN & CONTOURS

SHEET NO C3.2

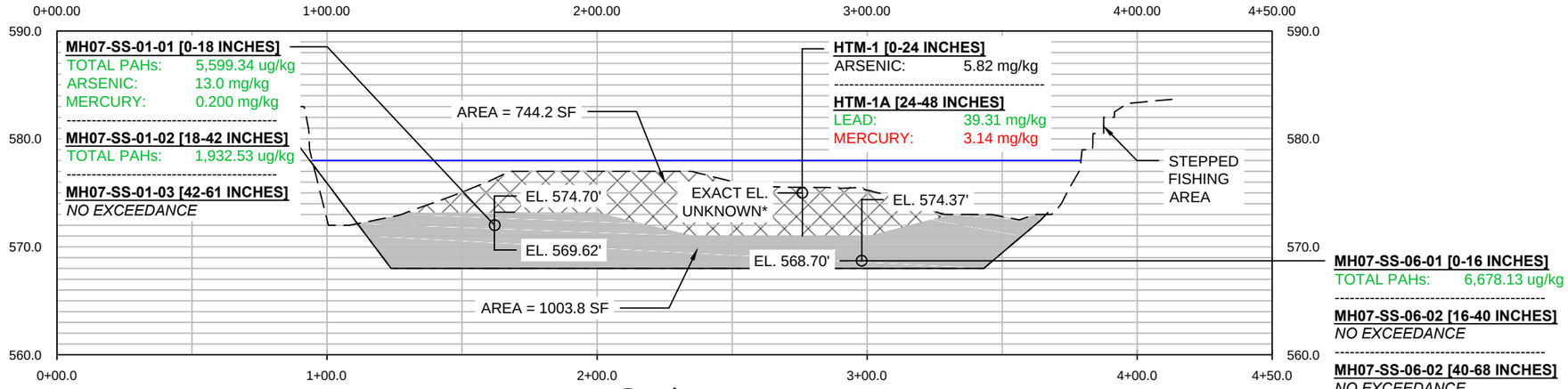
LEGEND:

- ENVIRONMENTAL DREDGE SPOILS (TO LANDFILL)
- NAVIGATIONAL DREDGE SPOILS (TO LOT 24/BENEFICIAL RE-USE)
- BENEFICIAL USE FILL
- EXISTING GRADE LINE
- DREDGE GRADE LINE
- FINAL GRADE LINE

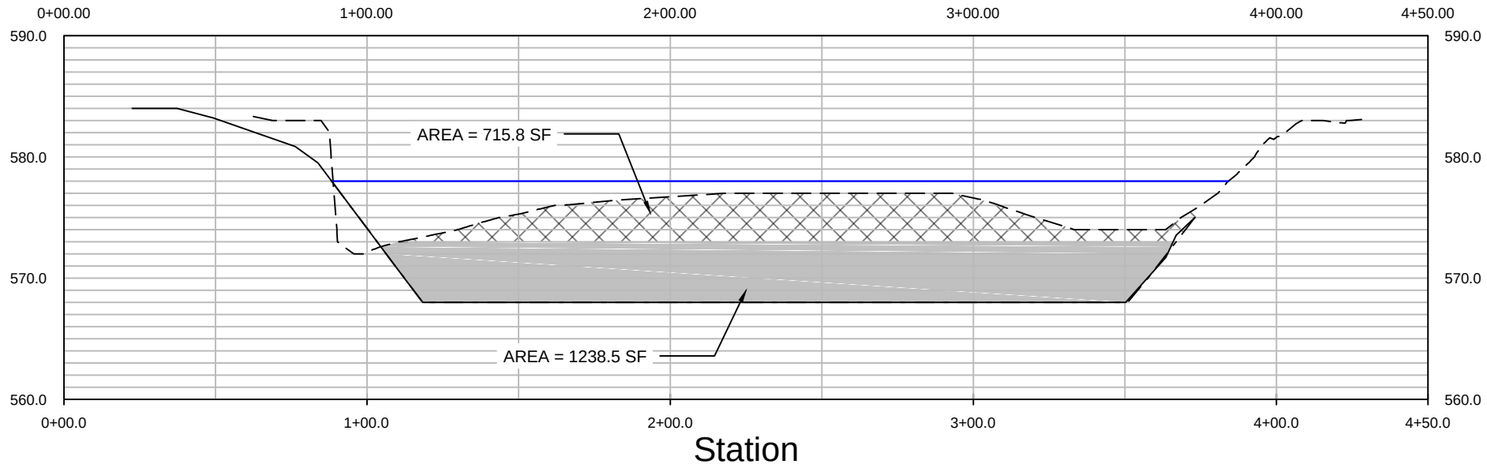
NOTES:

1. BORING LOCATIONS NOT LOCATED DIRECTLY ON CROSS SECTION LOCATION. INCONSISTENCIES IN GRADE LINES TOP OR BOTTOM OF SAMPLE MAY BE DUE TO LATERAL OFFSETS. SEE SHEET C2.1 FOR EXACT LOCATION OF SAMPLES.
2. GREEN TEXT INDICATES TEC EXCEEDENCE.
3. BLUE TEXT INDICATES TEC & MEC EXCEEDENCE.
4. RED TEXT INDICATES TEC, MEC, & PEC EXCEEDENCE.
5. * EXACT ELEVATIONS NOT RECORDED. ASSUMED AT TOP OF SILT AS SURVEYED NOVEMBER 2011 BY AYRES ASSOCIATES.
6. MAJORITY OF SEDIMENT IS SILTY SAND. WOOD DEBRIS ENCOUNTERED DURING SAMPLING AND ANTICIPATED THROUGHOUT HARBOR.
7. HARBOR WATER LEVEL ELEVATION SHOWN AT TIME OF SURVEY (EL. 578.0). DURING HIGH LAKE LEVEL CYCLE, HARBOR ELEVATION MAY REACH EL. 582.5.

Profile View of Cross-Section 1



Profile View of Cross-Section 2



DES BY G. SHAMBEAU	BOOK NO ----							MENEKAUNEE HARBOR IMPROVEMENTS CITY OF MARINETTE MARINETTE, WISCONSIN	AYRES ASSOCIATES EAU CLAIRE, WISCONSIN	HARBOR CROSS SECTIONS - 1	SHEET NO C4.0
DR BY G. SHAMBEAU	PROJ NO 19-0277.33										
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION				

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LEGEND:

- ENVIRONMENTAL DREDGE SPOILS
- NAVIGATIONAL DREDGE SPOILS
- BENEFICIAL USE FILL
- EXISTING GRADE LINE
- DREDGE GRADE LINE
- FINAL GRADE LINE

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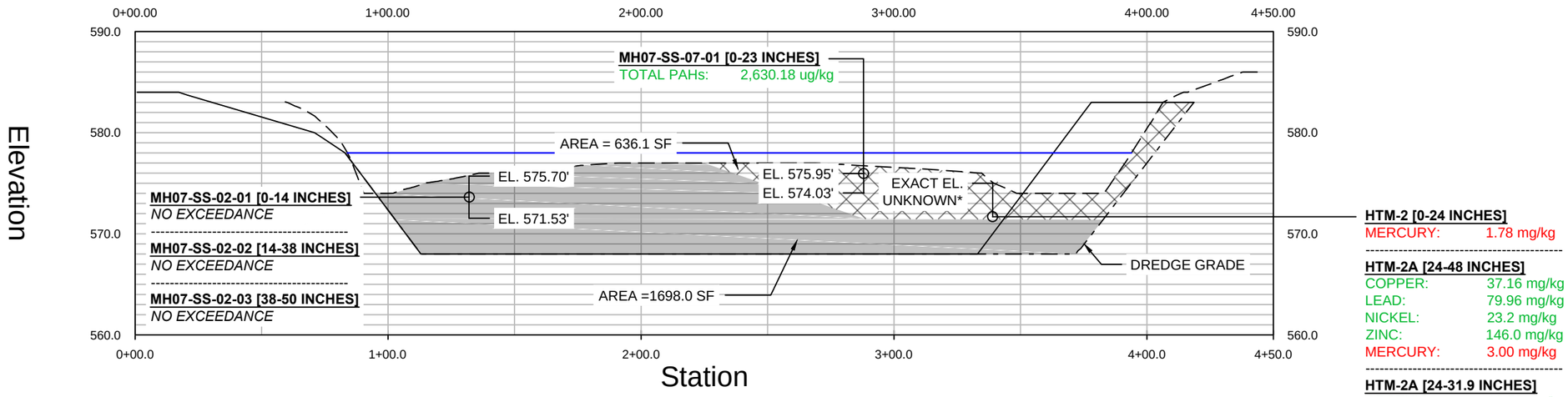
Profile View of Cross-Section 3



HTM-2 [0-24 INCHES]
MERCURY: 1.78 mg/kg

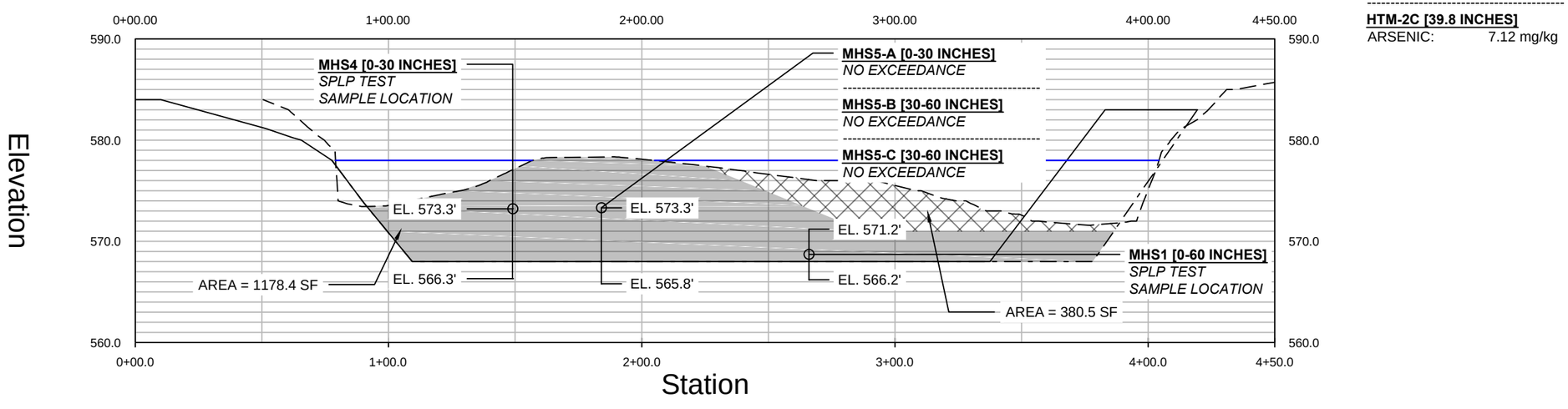
HTM-2A [24-48 INCHES]
COPPER: 37.16 mg/kg
LEAD: 79.96 mg/kg
NICKEL: 23.2 mg/kg
ZINC: 146.0 mg/kg
MERCURY: 3.00 mg/kg

HTM-2A [24-31.9 INCHES]
ARSENIC: 13.58 mg/kg

HTM-2B [31.9-39.8 INCHES]
ARSENIC: 5.26 mg/kg

HTM-2C [39.8 INCHES]
ARSENIC: 7.12 mg/kg

Profile View of Cross-Section 4



DES BY G. SHAMBEAU	BOOK NO ----						
DR BY G. SHAMBEAU	PROJ NO 19-0277.33						
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
EAU CLAIRE, WISCONSIN

HARBOR CROSS SECTIONS - 2

SHEET NO C4.1

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LEGEND:

- ENVIRONMENTAL DREDGE SPOILS
- NAVIGATIONAL DREDGE SPOILS
- BENEFICIAL USE FILL
- EXISTING GRADE LINE
- DREDGE GRADE LINE
- FINAL GRADE LINE

NOTES:

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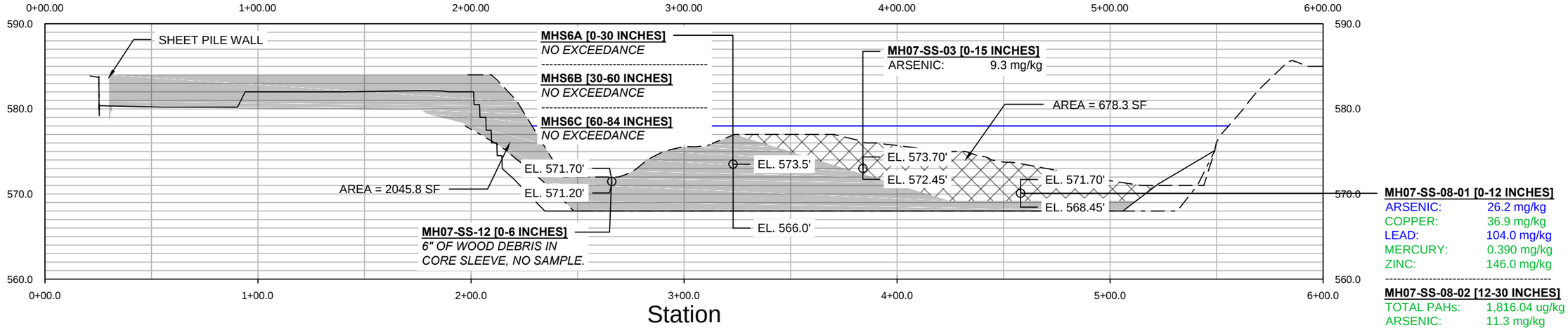
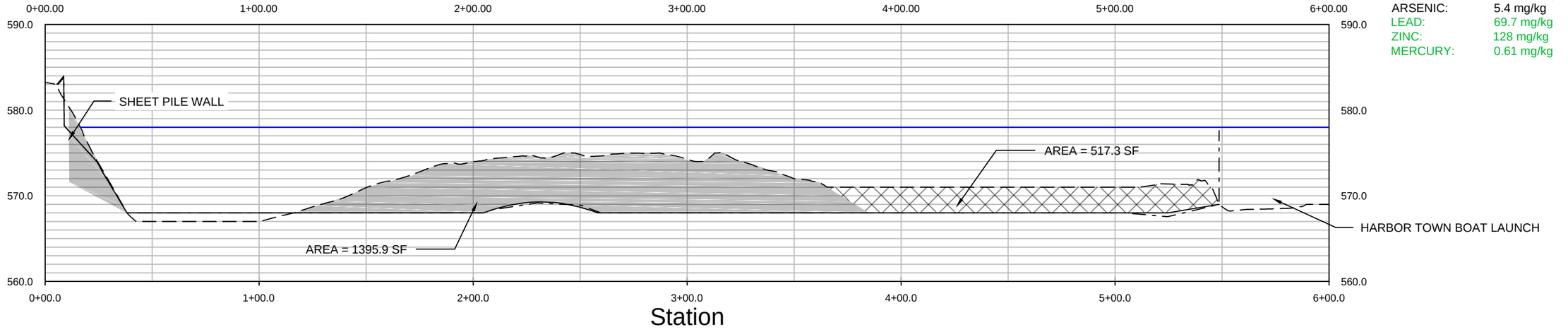
7. HARBOR WATER LEVEL ELEVATION SHOWN AT TIME OF SURVEY (EL. 578.0). DURING HIGH LAKE LEVEL CYCLE, HARBOR ELEVATION MAY REACH EL. 582.5.

Profile View of Cross-Section 5



Profile View of Cross-Section 6



DES BY G. SHAMBEAU	BOOK NO ----				MENEKAUNEE HARBOR IMPROVEMENTS CITY OF MARINETTE MARINETTE, WISCONSIN	AYRES ASSOCIATES EAU CLAIRE, WISCONSIN	SHEET NO C4.2
DR BY G. SHAMBEAU	PROJ NO 19-0277.33						
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION			HARBOR CROSS SECTIONS - 3

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LEGEND:

- ENVIRONMENTAL DREDGE SPOILS
- NAVIGATIONAL DREDGE SPOILS
- BENEFICIAL USE FILL
- EXISTING GRADE LINE
- DREDGE GRADE LINE
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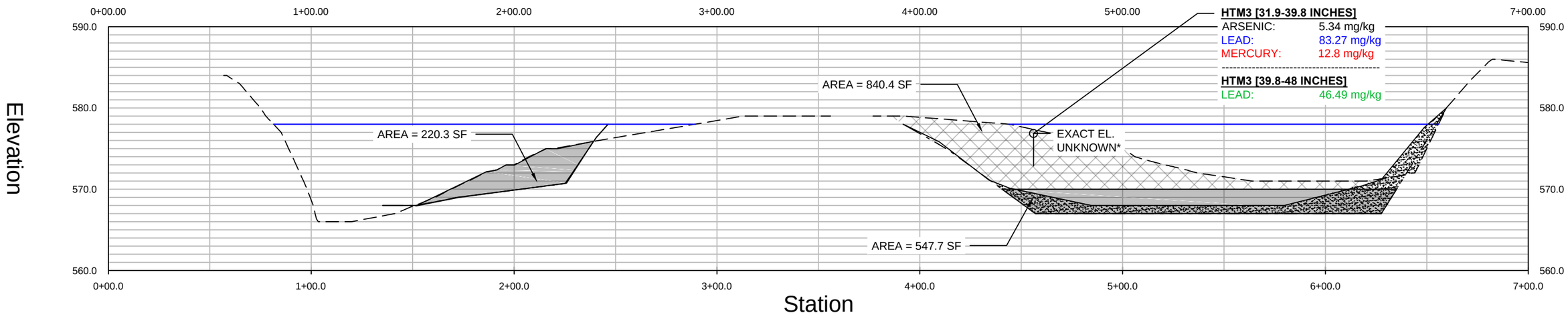
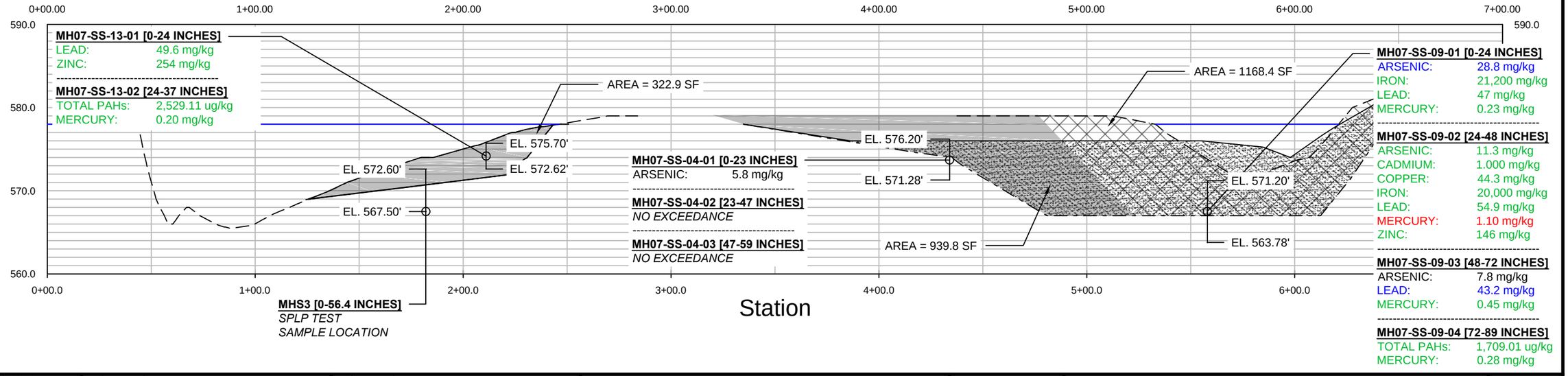
Profile View of Cross-Section 7



Profile View of Cross-Section 8



DES BY G. SHAMBEAU	BOOK NO ----						
DR BY G. SHAMBEAU	PROJ NO 19-0277.33						
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
EAU CLAIRE, WISCONSIN

HARBOR CROSS SECTIONS - 4

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LEGEND:

- ENVIRONMENTAL DREDGE SPOILS
- NAVIGATIONAL DREDGE SPOILS
- BENEFICIAL USE FILL
- EXISTING GRADE LINE
- DREDGE GRADE LINE
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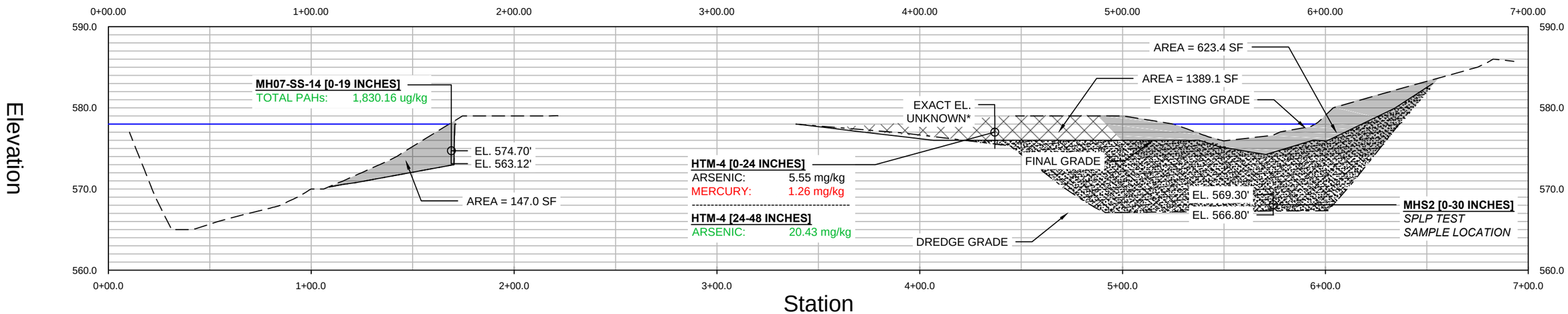
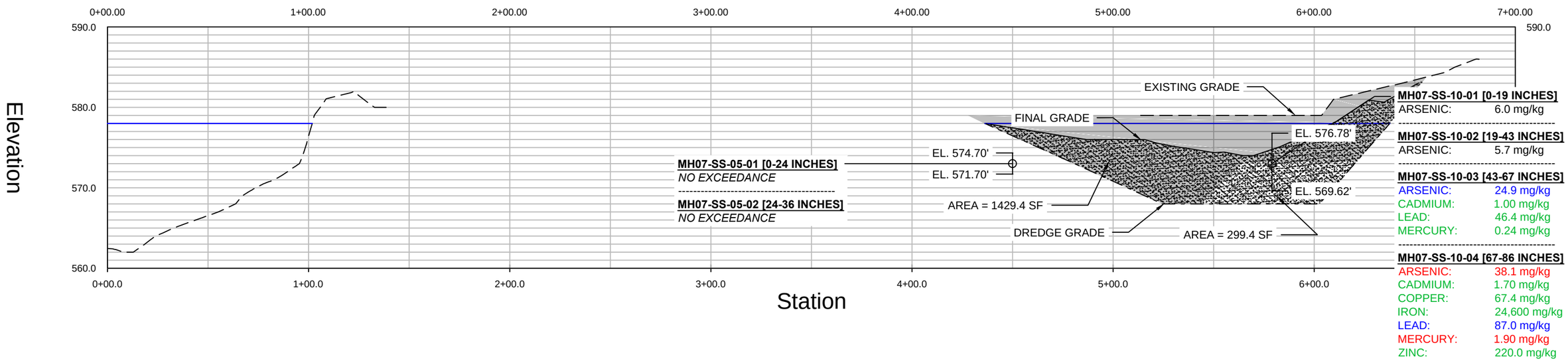
Profile View of Cross-Section 9



Profile View of Cross-Section 10



DES BY G. SHAMBEAU	BOOK NO ----						
DR BY G. SHAMBEAU	PROJ NO 19-0277.33						
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
EAU CLAIRE, WISCONSIN

HARBOR CROSS SECTIONS - 5

SHEET NO C4.4

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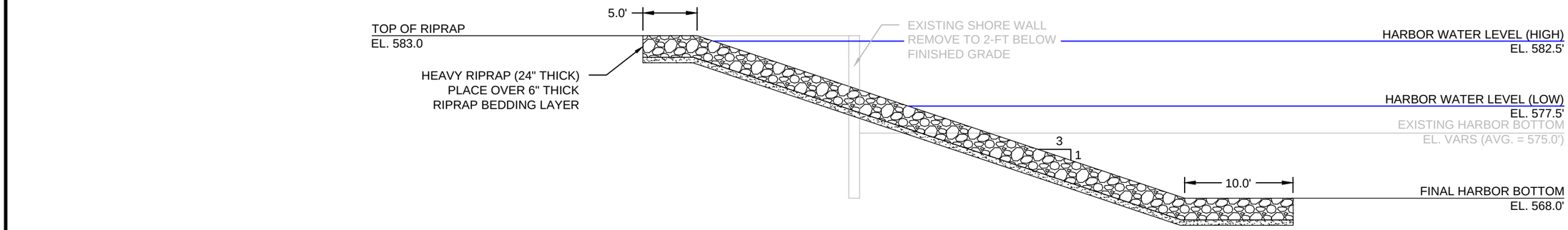
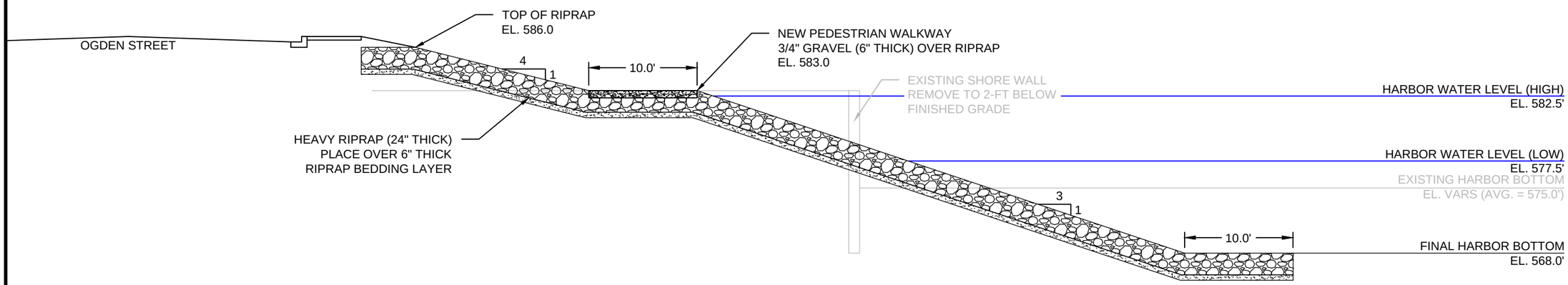
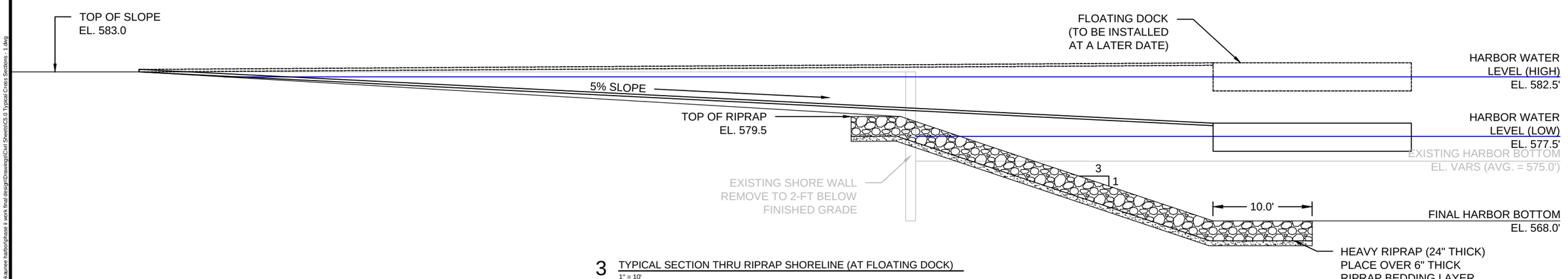


1 TYPICAL SECTION THRU RIPRAP SHORELINE
1" = 10'



2 TYPICAL SECTION THRU RIPRAP SHORELINE (ALONG OGDEN STREET)
1" = 10'



3 TYPICAL SECTION THRU RIPRAP SHORELINE (AT FLOATING DOCK)
1" = 10'

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6/5/2014
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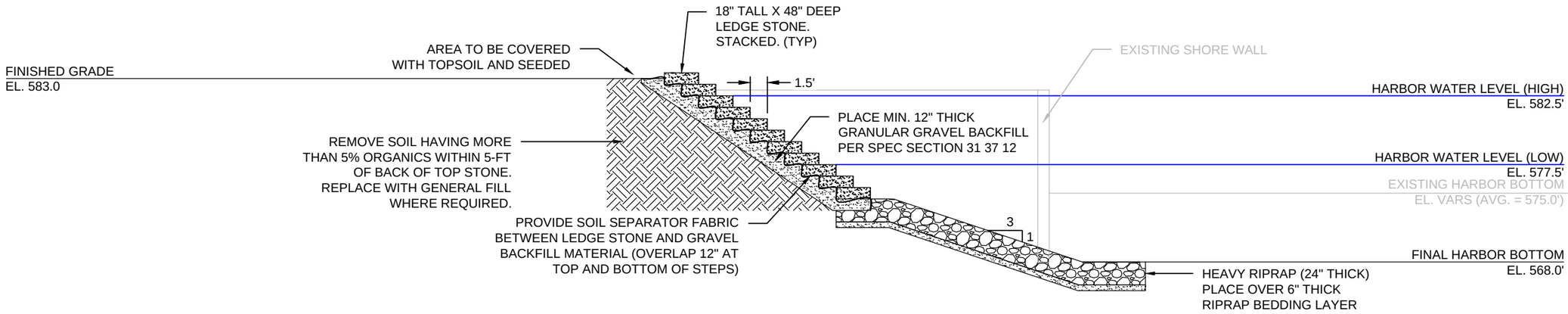
DES BY G. SHAMBEAU	BOOK NO ----						
DR BY G. SHAMBEAU	PROJ NO 19-0277.33						
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

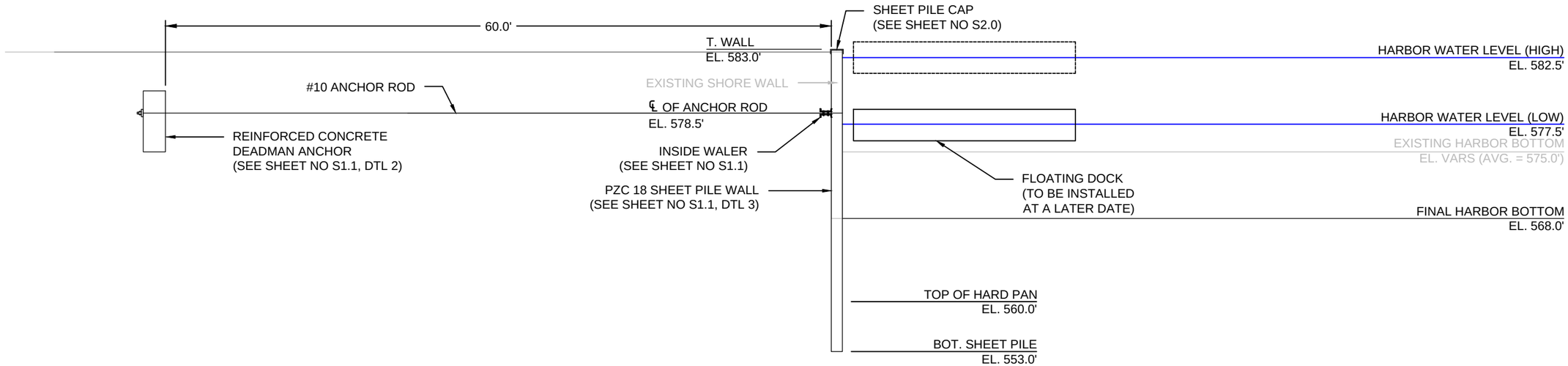
AYRES ASSOCIATES
EAU CLAIRE, WISCONSIN

TYPICAL CROSS SECTIONS - 1

SHEET NO
C5.0



1 TYPICAL CROSS SECTION THRU LEDGE STONE SHORELINE
1" = 10'



2 TYPICAL SECTION THRU SHEET PILE WALL
1" = 10'

GENERAL NOTES:

1. OVER-EXCAVATE ALL ENCOUNTERED VERY LOOSE SOILS (N<5 BPF) FROM WITHIN 3-FT OF THE BEARING ELEVATION AND 10-FT OF THE WALL SIDE OF THE CONCRETE DEADMAN ANCHOR.
2. REMOVE SOILS HAVING MORE THAN 5% ORGANICS FROM WITHIN 5-FT OF THE BEARING ELEVATION OF THE DEADMAN ANCHOR.

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k:\water resource eng\menekaunee harbor\phase ii work final design\Drawings\Civil Sheets\C5.1 Typical Cross Sections - 2.dwg

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DR BY G. SHAMBEAU	PROJ NO 19-0277.33								
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION		

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

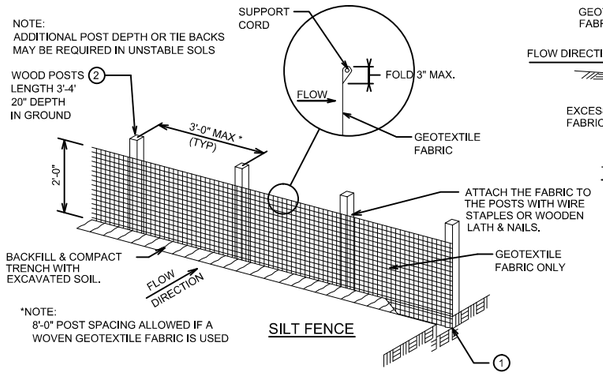
AYRES ASSOCIATES
EAU CLAIRE, WISCONSIN

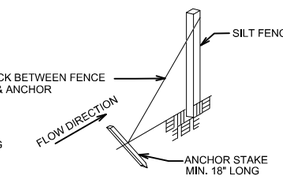
TYPICAL CROSS SECTIONS - 2

SHEET NO
C5.1

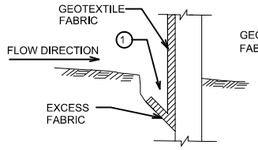
GENERAL NOTES:

1. TRENCH SHALL BE A MIN OF 4" WIDE & 6" DEEP TO BURY AND ANCHOR THE GEOTEXTILE FABRIC, FOLD THE MATERIAL TO FIT TRENCH AND BACKFILL & COMPACT TRENCH WITH EXCAVATED SOIL.
2. WOOD POSTS SHALL BE A MIN SIZE OF 1 ½" x 1 ½" OAK OR HICKORY.
3. CONSTRUCT SILT FENCE FROM A CONTINUOUS ROLL IF POSSIBLE BY CUTTING LENGTHS TO AVOID JOINTS. IF A JOINT IS NECESSARY, USE ONE OF THE FOLLOWING TWO METHODS;
   A. TWIST METHOD -- OVERLAP THE END POSTS AND TWIST, OR ROTATE, AT LEAST 180 DEGREES.
   B. HOOK METHOD -- HOOK THE END OF EACH SILT FENCE LENGTH.

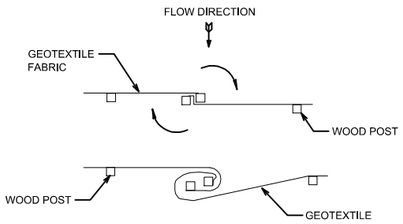




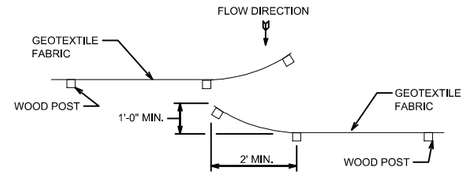
SILT FENCE TIE BACK
(WHEN ADDITIONAL SUPPORT REQUIRED)



TRENCH DETAIL



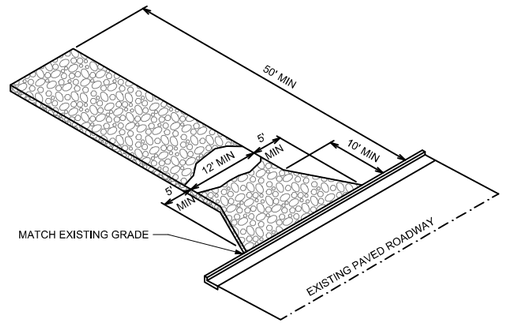
TWIST METHOD



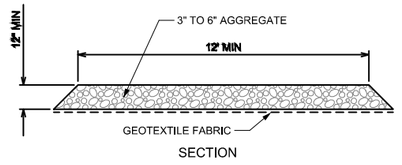
HOOK METHOD

JOINING 2 LENGTHS OF SILT FENCE 3

1 SILT FENCE DETAIL
0156201

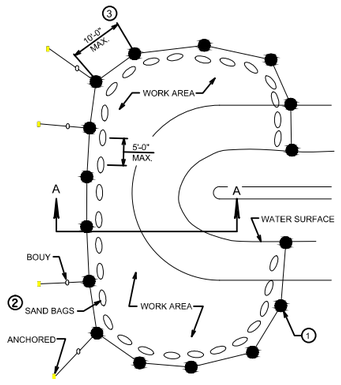


PLAN

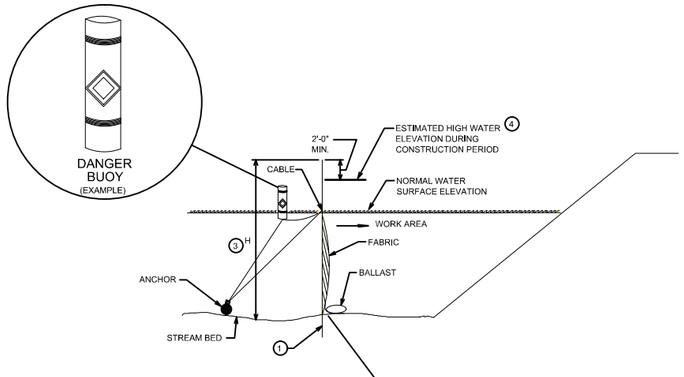


SECTION

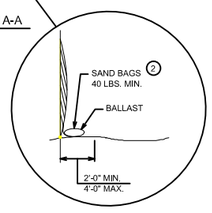
2 STONE TRACKING PAD
0156201



PLAN VIEW (TYPICAL)



SECTION A-A



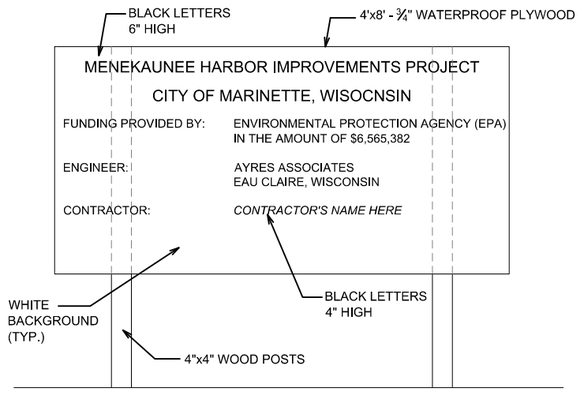
NOTES:

1. DRIVEN STEEL POSTS. PIPES, OR CHANNELS. LENGTH SHALL BE SUFFICIENT TO SECURELY SUPPORT BARRIER AT HIGH WATER ELEVATIONS.
2. SAND BAGS TO BE USED AS ADDITIONAL BALLAST WHEN ORDERED BY THE ENGINEER TO MEET ADVERSE FIELD CONDITIONS.
3. WHEN BARRIER HEIGHT, H, EXCEEDS 8'-0" POST SPACING MAY NEED TO BE DECREASED.
4. ELEVATION VALUE TO BE ESTABLISHED BY THE CONTRACTOR BASED ON THE TIME OF YEAR AND DURATION OF THE ACTIVITY.
5. TURBIDITY BARRIER SHALL COMPLETELY ENCLOSE ACTIVE DREDGING AREA AND BREAKWATER CONSTRUCTION AREA. CONTRACTOR SHALL DETERMINE LENGTH OF TURBIDITY BARRIER REQUIRED FOR CONTRACTORS OPERATION.

3 TURBIDITY BARRIER DETAIL
N.T.S 0156203

NOTES:

1. ALL PAINT SHALL BE EXTERIOR GRADE ENAMEL IN COLOR INDICATED.
2. SIGN SHOULD INCLUDE GREAT LAKES RESTORATION INITIATIVE LOGO (GLRI) IN LOWER RIGHT HAND CORNER OF SIGN. GLRI LOGO TO BE PROVIDED TO CONTRACTOR BY EPA.
3. FINAL SIGN DETAILS AND INFORMATION TO BE COORDINATED WITH OWNER.



4 PROJECT INFORMATION SIGN

DES BY G. SHAMBEAU	BOOK NO ----							
DR BY G. SHAMBEAU	PROJ NO 19-0277.33							
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION	

MENEKAUNEE HARBOR IMPROVEMENTS
CITY OF MARINETTE
MARINETTE, WISCONSIN

AYRES ASSOCIATES
EAU CLAIRE, WISCONSIN

MISC. SITE DETAILS

SHEET NO C6.0

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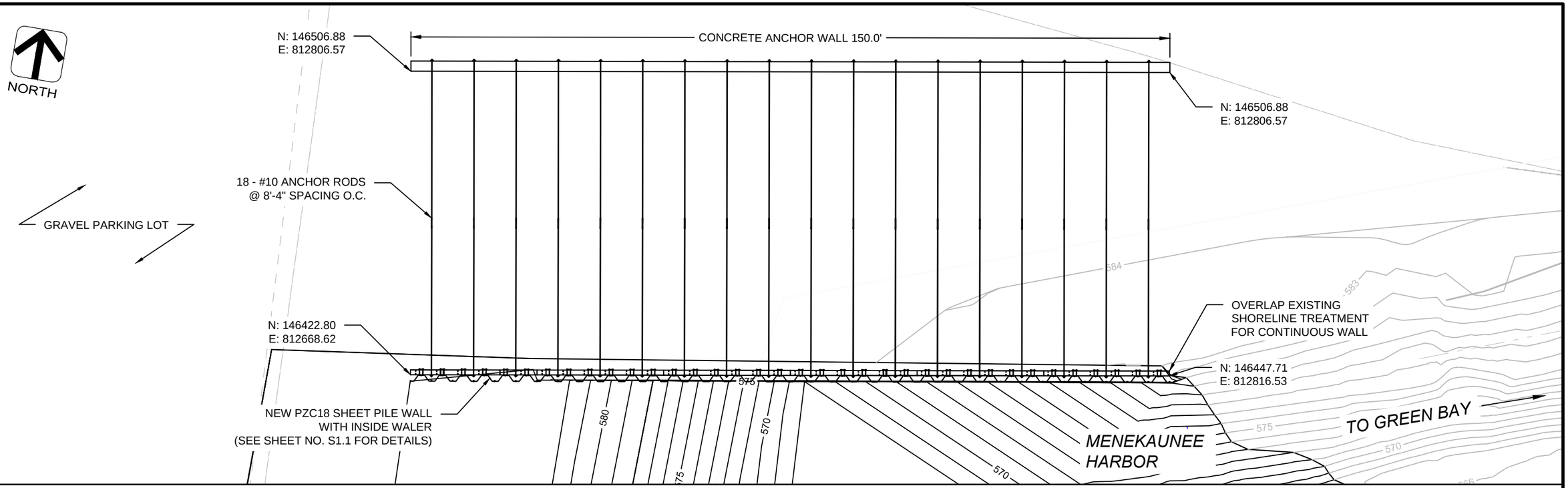
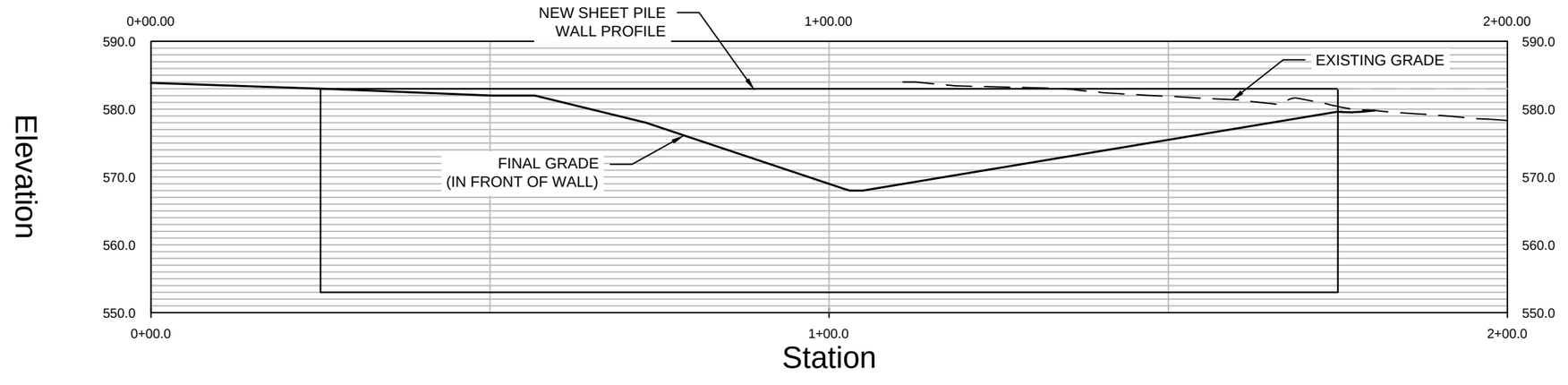


Profile View of Sheet Pile Wall



20 10 0 20 40FT

SCALE IN FEET

DES BY G. SHAMBEAU	BOOK NO ----	NO	DATE	REVISION	NO	DATE	REVISION	MENEKAUNEE HARBOR IMPROVEMENTS CITY OF MARINETTE MARINETTE, WISCONSIN	AYRES ASSOCIATES EAU CLAIRE, WISCONSIN	SHEET PILE WALL PLAN & PROFILE	SHEET NO S1.0
DR BY G. SHAMBEAU	PROJ NO 19-0277.33										
CHK BY C. GOODWIN	DATE JUNE 2014										

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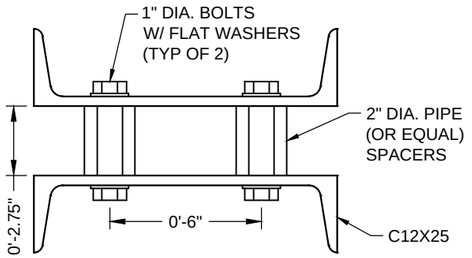
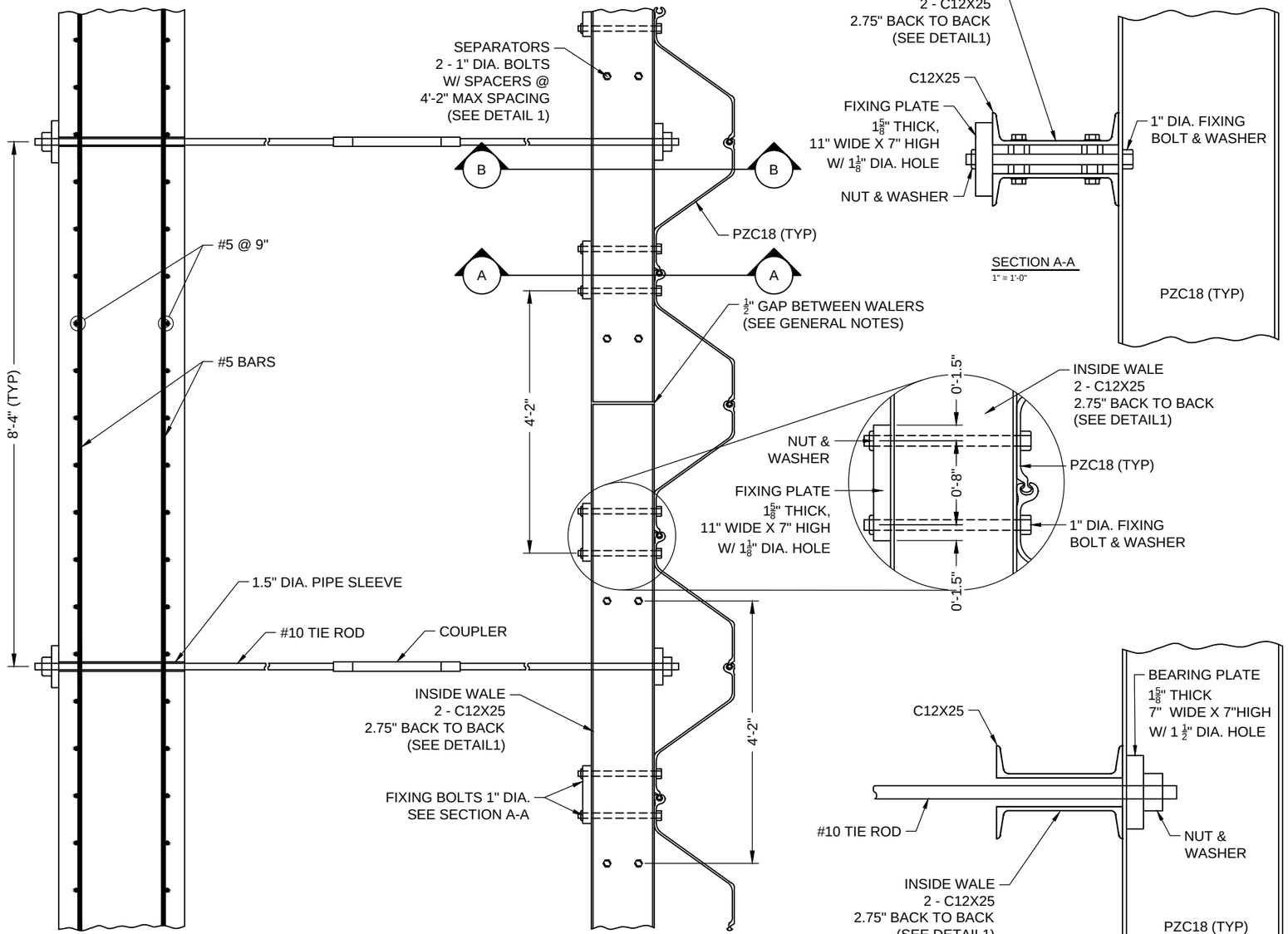
GENERAL NOTES:

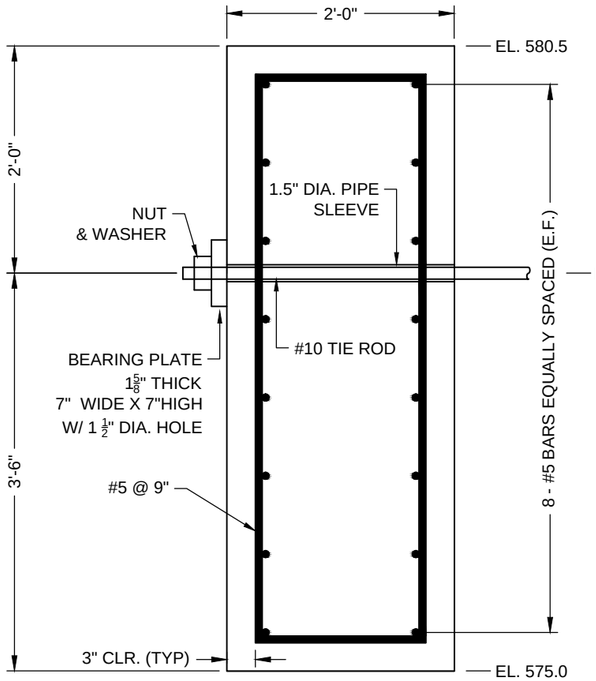
1. GAP BETWEEN WALERS TO BE LOCATED HALF WAY BETWEEN ADJACENT TIE RODS.
2. EACH WALER SECTION SHALL SPAN A MINIMUM OF TWO TIE RODS (MINIMUM LENGTH = 16'-8").



1 SEPARATOR DETAIL
2" = 1'-0"



SECTION A-A
1" = 1'-0"



2 SECTION THRU CONCRETE DEADMAN ANCHOR
3/4" = 1'-0"

SECTION B-B
1" = 1'-0"

3 TYPICAL SECTION THRU SHEET PILE WALL
1/2" = 1'-0"

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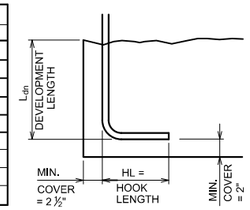
DR BY G. SHAMBEAU	PROJ NO 19-0277.33									
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION			

BAR SIZE GRADE 60	f'_c = 4000 psi	
	L_{dh}	HL
#3	6"	6"
#4	7"	8"
#5	9"	10"
#6	10"	1'-0"
#7	1'-0"	1'-2"
#8	1'-2"	1'-4"
#9	1'-3"	1'-8"
#10	1'-5"	1'-10"
#11	1'-7"	2'-0"



COMPLYING WITH MINIMUM COVER REQUIREMENTS OF ACI 318 12.5 OTHERWISE L_{dh} MUST BE RE-CALCULATED

1 TYPICAL 90 DEGREE HOOK FOR REBAR

0512002

MINIMUM DEVELOPMENT AND SPLICE LENGTHS

f'_c = 4000 psi
clear spacing $\geq 2d_b$ & clear cover $\geq d_b$

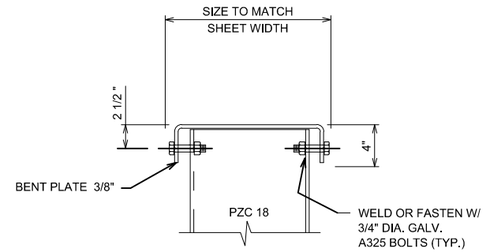
Bar Size Grade 60	Minimum Straight Tension Development Length, l_d		Minimum Lap Splice For Center To Center Bar Spacing	
	TOP BARS	OTHER BARS	TOP BARS	OTHER BARS
#3	1'-7"	1'-3"	2'-0"	1'-7"
#4	2'-1"	1'-7"	2'-9"	2'-1"
#5	2'-7"	2'-0"	3'-5"	2'-7"
#6	3'-1"	2'-5"	4'-1"	3'-1"
#7	4'-6"	3'-6"	5'-11"	4'-6"
#8	5'-2"	4'-0"	6'-9"	5'-2"
#9	5'-10"	4'-6"	7'-7"	5'-10"
#10	6'-7"	5'-1"	8'-6"	6'-7"
#11	7'-3"	5'-7"	9'-5"	7'-3"

NOTES:

1. TOP BARS ARE HORIZONTAL BARS SO PLACED THAT MORE THAN 12" OF CONCRETE IS CAST IN THE MEMBER BELOW THE BAR.
2. EXCEPT AS OTHERWISE INDICATED ON THE DRAWINGS, LAP SPLICE LENGTH AND TENSION DEVELOPMENT l_d SHALL BE NO LESS THAN (NO MINUS TOLERANCE) SHOWN ABOVE.
3. LAP SPLICES SHALL NOT BE MADE AT POINTS OF MAXIMUM STRESS AS DETERMINED BY THE ENGINEER.
4. WHERE BARS OF DIFFERENT SIZE ARE LAP SPLICED, SPLICE LENGTH SHALL BE THE SPLICE LENGTH OF THE LARGER BAR.
5. ALL SPLICES SHALL BE CONTACT SPLICES AND WIRED TOGETHER.

2 MINIMUM DEVELOPEMENT & SPLICE LENGTH FOR REBARS

0512001



3 SECTION THRU SHEET PILE CAP

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DES BY G. SHAMBEAU	BOOK NO ----						MENEKAUNEE HARBOR IMPROVEMENTS CITY OF MARINETTE MARINETTE, WISCONSIN	AYRES ASSOCIATES EAU CLAIRE, WISCONSIN	STRUCTURAL DETAILS	SHEET NO S2.0
DR BY G. SHAMBEAU	PROJ NO 19-0277.33									
CHK BY C. GOODWIN	DATE JUNE 2014	NO	DATE	REVISION	NO	DATE	REVISION			

APPENDIX B

Sevenson Work & Dewatering Plan

WORK & DEWATERING PLAN

MENEKAUNEE HARBOR IMPROVEMENTS
MARINETTE, WISCONSIN

CITY OF MARINETTE

Submitted by:

Sevenson Environmental Services, Inc.

NIAGARA FALLS, NEW YORK

1 INTRODUCTION

Sevenson Environmental Services, Inc. (SES) presents this plan to outline the means and methods that will be implemented to perform dredging and dewatering operations within the Menekaunee Harbor Improvements Project.

This work and dewatering plans below have been developed as required by Section 35 20 13 - Dredging of the contract documents.

2 WORK PLAN

2.1 DREDGING SCHEDULE

Sevenson plans to start onsite activities as soon as NTP is provided by the City of Marinette. A detailed project schedule will be provided as a separate submittal. As it pertains to dredging, removal of sediments will commence as soon as the dewatering pad/work area site preparation is completed.

Based on the information provided, Sevenson anticipates the following production rates for dredging:

Placement Area	Method	Avg Dredging Rate	Quantity	Time (Work Days)
Lot 24	Mechanical	800 CY/Day	25,000 CY	32
Landfill	Mechanical	800 CY/Day	27,500 CY	35
Beneficial Reuse	Hydraulic	500 CY/Day	22,500 CY	45

Sevenson plans to begin mechanical dredging operations in the area which receives beneficial reuse material immediately. Once this area is excavated, the hydraulic excavator may begin working simultaneously within the harbor.

2.2 DREDGING METHODS

Sevenson's plan is to utilize both mechanical and hydraulic dredging for this project. Hydraulic dredging will be utilized for the dredging and direct hydraulic placement of the beneficial reuse fill material. Hydraulic dredging and direct placement provides greater flexibility and more controllable method for distributing the fill in the beneficial reuse area.

The material to be transported offsite (Lot 24 and the WM Landfill) will be dredged mechanically via excavator on a barge. The excavator will load smaller material scows, which will be transported via work boat to the landside work area where another excavator will offload the dredged material. The mechanical excavation significantly reduces the quantity of water to be handled throughout the project.

2.2.1 Equipment

Mechanical Dredge – Caterpillar 345 Hydraulic Excavator

- Spud Barge Dimensions: 40-feet x 60-feet x 5-feet
- Bucket: 3 cubic yard level-cut hydraulic closing bucket



Figure 1 – Cat 345 with Hydraulic Bucket Loading a 100 cy Material Scow

Hydraulic Dredge – 8–inch-diameter Dredge "Moray"

- Overall dredge length: 42-feet including ladder in the up position
- Overall dredge width: 11-feet
- Hull dimensions (excluding ladder): length 35-feet (42-feet with ladder up), width 11-feet, depth 4-feet, draft 1.5-feet
- Dredge can operate in swinging ladder mode or conventional mode
- Cutter diameter: 28-inches
- Hospital grade muffler installed for added noise reduction



Figure 2 – 8" Moray Dredge

Attendant Plant

- 100 CY Material Barges (3)
- Work Boats (2 – 150hp, 1 – 25hp)

2.2.2 Collecting & Treating Water

See Section 3 Dewatering Plan.

2.2.3 Dredging Accuracy

Each dredge plant will be equipped with Hypack Dredgepack dredge positioning software with DGPS antenna. The existing dredge surface and target dredge surfaces will be programmed into the system. The operator will simultaneously view a plan and profile of the dredge cut on the computer screen. The hydraulic dredge and mechanical excavator are outfitted with inclinometers and other positioning equipment which works in sequence with the mounted DGPS and software to provide the operator with a precise position (within 0.2 feet) of the bucket in three dimensional space. The positioning data and operator input is utilized to track performance and production and provide operational guide to the operator. This positioning system will exceed the specified horizontal and vertical dredging tolerance requirements, which will minimize any over or under dredging.

Shoreline structures will also be programmed into the software and visually flagged where appropriate to guide the dredge operator. An approximate buffer between any critical structure and the dredging operations will be discussed and reviewed with the owner's representative.

2.2.4 Beneficial Reuse Placement

The material to be used within the harbor for beneficial reuse will be dredged and placed hydraulically within the designated areas and to the lines and grades shown on the contract drawings. Sevenson plans to

utilize various inflow points coupled with hydrographic surveying and inspection to monitor, assess, and control the placement and turbidity of dredged sediments in the beneficial reuse area.

Sevenson will utilize the hydraulically pumped slurry's angle of repose to distribute material across the areas and limit mechanical manipulation. The beneficial reuse material is very granular (>90%) and contains only a small fraction of fine grained sediments. This material is not known to cause significant turbidity issues. In addition to the turbidity curtains at the entrance of the harbor, Sevenson also plans to also utilize an impermeable silt curtain across the majority of beneficial reuse placement area.

Best management practices, such as reducing the discharge rate, modifying the discharge position/elevation, and or increasing/decreasing the slurry's percent solids; will be implemented should turbidity or resuspension of existing sediments become a concern.

2.3 SEQUENCING

As detailed, in the schedule, Sevenson will initiate dredging activities in the area which receives beneficial reuse material. The mechanical dredging in this area includes both Lot 24 (navigation) and Landfill (environmental) sediments. Dredge operators will have their software set up to perform only one sediment type at a time, to avoid cross contamination.

Dredging operations are tentatively scheduled to follow the lines and grades as shown on the contract drawing cross sections. Pre-dredging in-situ samples collected for the beneficial reuse materials may alter the plan based on metals concentrations from the lab testing.

Upon completion of the dredging within the beneficial reuse area, the mechanical dredging operation will focus on shoreline dredging work to permit shoreline treatment activities to commence (ledge stone, armoring). The hydraulic dredge will focus on dredging and placement of beneficial reuse material.

2.4 EROSION CONTROL & SECURITY DETAILS

Sevenson will provide curbing around the dewatering pad and erosion controls as detailed on the site drawings. Temporary fencing will be used as needed to restrict access to the site.

2.5 WATERWAY MARKERS, BOAT TRAFFIC CONTROL, AND PROTECTION OF COMMERCIAL/RECREATIONAL WATERCRAFT

Sevenson will provide temporary private aids to navigation I and markers as detailed in the Waterway Marker Permit Guidelines. All aids to navigation will be inspected on a daily basis to ensure proper operation. Sevenson will repair and replace all aids to navigation as necessary. These lighted boys will be station along the proposed silt curtain alignment to clearly mark the alignment and work zone.

For the duration of the project, Sevenson will monitor navigation activities within the Menekaunee Harbor. For proper monitoring of marine traffic and all marine-based project activities, Sevenson will use combinations of the following maritime devices (when applicable):

- Marine VHF Radios
- Navigation-safety equipment (i.e., depth sounding equipment, compass, radar, etc.)
- Vessel inspections of Sevenson equipment and subcontractor equipment.

The VHF radios will be capable of clearly receiving and transmitting radio communications over the Project Area. Sevenson will use a separate VHF channel for all job specific communication and will monitor VFH channels 13 and 16 for general marine communication.

All dredging and related activities will be within the Menekaunee Harbor. It is anticipated that no commercial traffic and little to no recreational traffic will need access to the project area. Should commercial traffic need to access the project area, move equipment and silt curtains as direct by the Engineer.

3 DEWATERING PLAN

Sevenson's dredging methodologies, for the dredged material to be placed off site, eliminates the abundance of water added via a hydraulic dredging. Mechanical dredging may add approximately 10% more water to the in-situ volume during mechanical dredging versus 3 to 5x the in-situ volume with hydraulic dredging. After review of provided data and similar work experiences, Sevenson anticipates that the dredged material will naturally dewater on-site to meet the project requirements in a very timely manner. Mechanically offloaded material will be placed, worked, and blended on a staging area pad to promote natural dewatering. The water will be directed and collected by installed sumps, positioned around the dewatering site.

3.1 DEWATERING SYSTEM DESIGN & PLANS

Mechanically dredged materials will be offloaded from material scows by a hydraulic excavator. Materials will be placed on a pad and segregated as needed to keep Lot 24 materials and Landfill material from mixing. The material will be worked on the pad with loaders and excavators to promote natural drainage. Once material meets the requirements for transport it will be either stored on-site or directly loaded into trucks for transport.

The collected water will be pumped directly to Sevenson's on-site water treatment system, prior to discharge to the local WWTP. Prior to discharging to the WWTP sewer connection, Sevenson will collect site and interstitial water from the dredged material and route this water to a series of onsite frac tanks. The

frac tanks will be utilized for initial settlement of suspended solids. From the frac tanks, water will be routed through a bag filtration system, prior to discharge through a flowmeter to a WWTP sewer. Sevenson will test the system to confirm that all operational constraints and WWTP requirements are being meant prior to beginning dredging activities. Our water treatment experience, in-house lab, and company owned equipment uniquely enable Sevenson to assess, analyze, and rapidly deploy additional equipment should the planned system experience any issues or not meet project requirements during testing or throughout the project duration.

3.2 PUMP INFORMATION

Pumping from the dewatering pad influent equalization tanks will be done with a Dri-Prime type pump. This pump will deliver process water to the Bag Filter unit via a 3" hose coupled with "Cam-Lock" quick disconnects. The pump cycle times and flow rate will be determined by the operator in charge of the Water Treatment System (WTS).

3.3 PIPING LAYOUT

A Layout Diagram has been attached to this document (Attachment 1) to illustrate the piping design of the WTS.

3.4 INSTRUMENTATION

The instrumentation for the WTS will consist of, at a minimum: Audible/Visual level alarms on the influent equalization tank(s) and a Rate/Totalizing flow meter on the discharge side of the final filter.

3.5 EQUIPMENT

Equipment for the WTS will be as follows:

- **Influent Equalization Tank (s) –** 21,000 gallon frac tanks to receive water from the De-Watering Sumps.
- **Pumps**- Two (2) Dri-Prime type pumps will be utilized to pump water from the Inf. /Eq. tanks through the Bag Filter Unit. Pumps will be piped in a "primary/standby" configuration.
- **Bag Filter Unit**- a Fil-Trek ELPA24, 6 element bag filter housing will be used to filter solids from the process water. Filter bag micron sizes will be determined on site.
- **Flow Meter** - An approved Flow Meter will be installed downstream of the Bag Filter Unit. Typically this meter will have rate and totalizing display.
- **Hoses** - 3", Versiflo, 150psi, S&D Rubber hard hose will be used to transfer The water from the Inf/EQ tanks to the Pump and then through the Flow Meter. Hoses will be secured to equipment using Cam-Lock connectors and stainless steel banding.



Figure 1 – Frac Tank and Bag Filtration Unit

3.6 CHEMICAL ADDITION

At this time, chemical additives such as flocculants and coagulants are not anticipated.

3.7 ANTICIPATED FLOW RATES

The water treatment system as designed will have a flow rate window of operation between 50-200 gallons per minute.

3.8 SYSTEM MONITORING

The water treatment system operations will be monitored by an operator who will be recording daily operations in a site specific WTS log book.

3.9 SAMPLING & TESTING PLAN

All water collected will be discharged to the City of Marinette's WWTP. Therefor our sampling and testing protocol matches the requirements of the Marinette Water Utility's letter, dated March 25, 2014. The plan is detailed below.

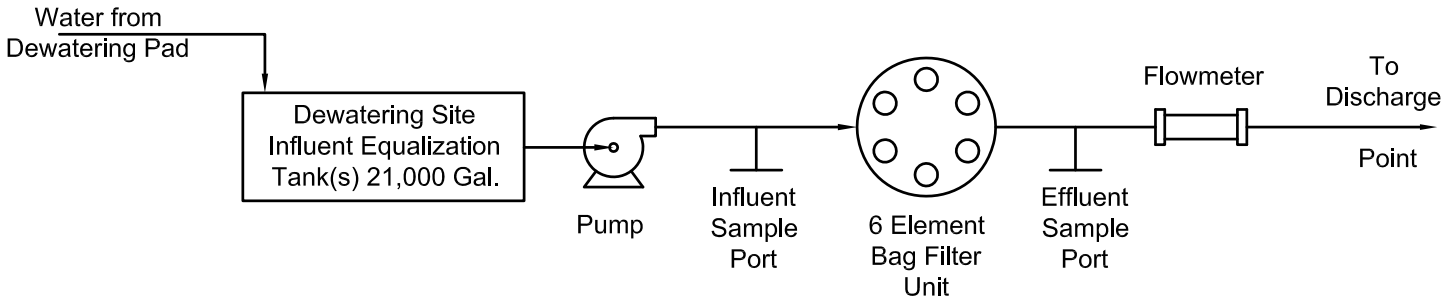
- Sampling & Testing Frequency
  - 1x Prior to 1st Discharge
  - 2x during Week 1
  - 1x per week during Weeks 2-5
  - 1x per month after Week 5

- Testing Parameters

Table 1: City of Marinette Wastewater Treatment Plant Local Limits

METALS		
Arsenic	mg/L	0.10
Cadmium	mg/L	0.14
Chromium	mg/L	2.41
Copper	mg/L	1.40
Lead	mg/L	2.02
Mercury	mg/L	0.01
Nickel	mg/L	1.35
Selenium	mg/L	0.12
Silver	mg/L	0.50
Zinc	mg/L	2.25
GRAB SAMPLES		
Cyanide	mg/L	0.19
Oil & Grease	mg/L	100
Phenols	mg/L	25
pH		6.0-9.0
SURCHARGEABLES		
BOD	mg/l	300
TSS	mg/l	350
PHOS	mg/l	14.5

Attachment 1



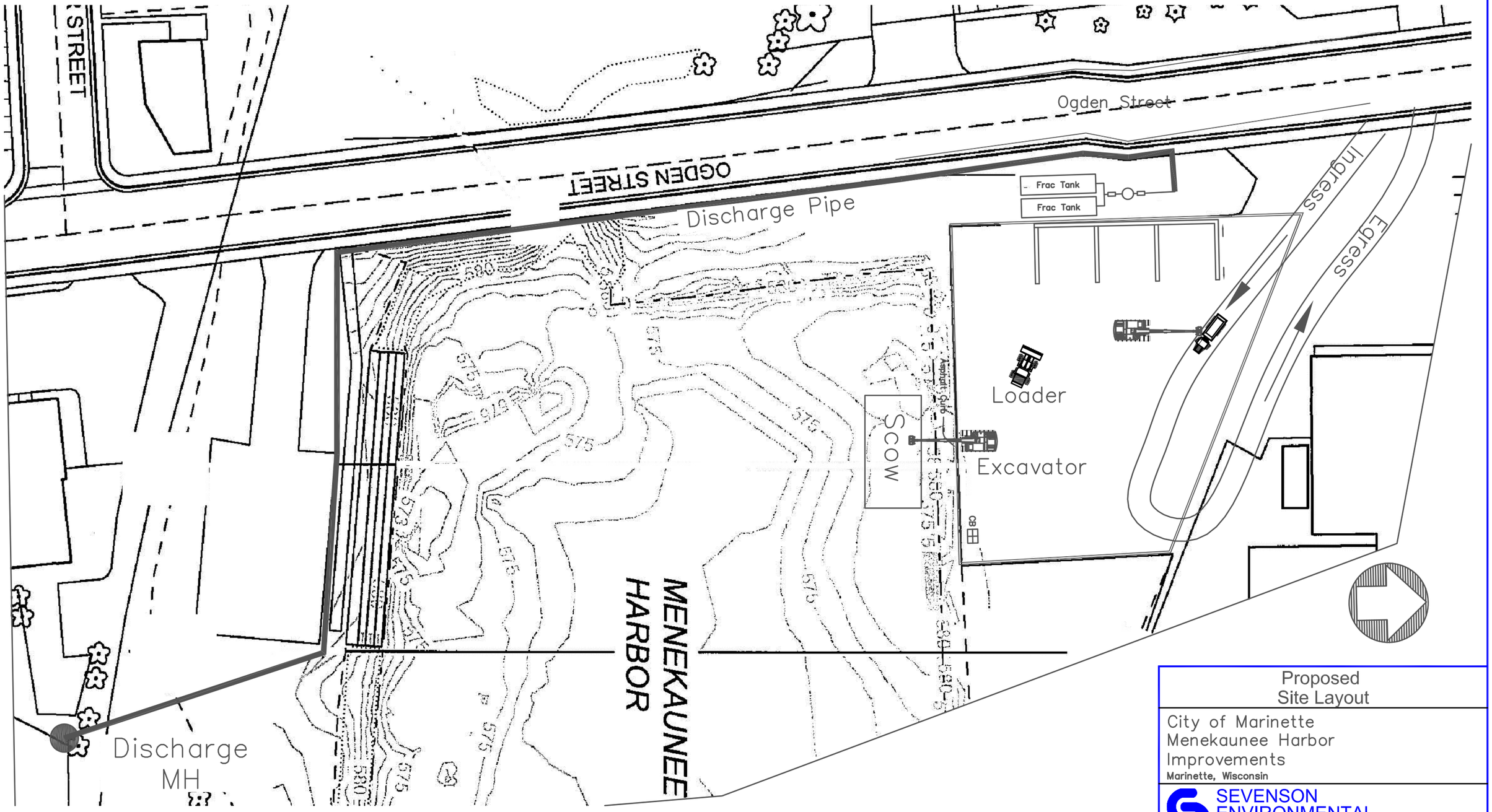
200 GPM Wastewater Treatment Process Flow Diagram

City of Marinette
Menekaunee Harbor
Improvements
Marinette, Wisconsin

SEVENSON ENVIRONMENTAL SERVICES, INC.

Figure
3

DATE:	8/4/14
DRAWN BY:	C. Bigelow
CHECKED BY:	M. Walker/ R. Elia, Jr.
CAD FILE:	wwtp
SCALE:	None



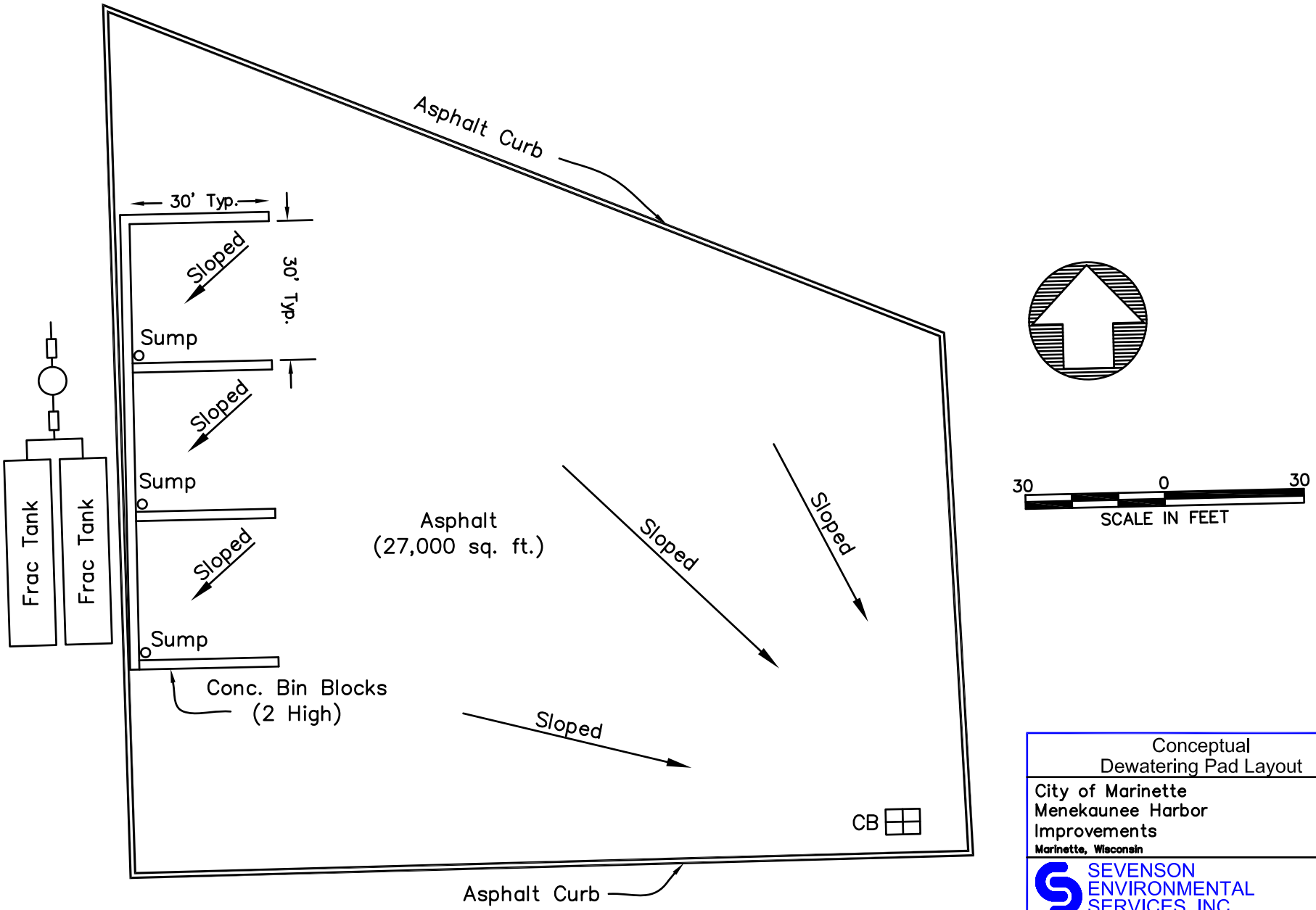
Proposed
Site Layout

City of Marinette
Menekaunee Harbor
Improvements
Marinette, Wisconsin

SEVENSON ENVIRONMENTAL SERVICES, INC.

DRAWING
1

DATE:	8/4/14
DRAWN BY:	C. Bigelow
CHECKED BY:	M. Walker/R. Elia, Jr.
CAD FILE:	Site Layout
SCALE:	As Shown



Conceptual
Dewatering Pad Layout

City of Marinette
Menekaunee Harbor
Improvements
Marinette, Wisconsin

SEVENSON
ENVIRONMENTAL
SERVICES, INC.

DRAWING
2

DATE:	8/1/14
DRAWN BY:	C. Bigelow
CHECKED BY:	M. Walker/R. Elia, Jr.
CAD FILE:	Dewater Site Layout
SCALE:	As Shown

SUPPLEMENT TO:
WORK & DEWATERING PLAN

MENEKAUNEE HARBOR IMPROVEMENTS
MARINETTE, WISCONSIN

CITY OF MARINETTE

Submitted by:

Sevenson Environmental Services, Inc.

NIAGARA FALLS, NEW YORK

1 INTRODUCTION

As per discussion and request on August 8, 2014, Sevenson Environmental Services, Inc. (SES) has prepared the following dredging sequence and schedule as a supplement to our Work & Dewatering Plan (submitted for approval by Mark Schmidt via email on 8/5/14).

2 SCHEDULE & PLAN VIEW LAYOUT

The following schedule and figures provides the general sequence of work SES will utilize to guide dredging operations in the field. Please note that the quantities and plan view areas are not exact. Utilizing the in-situ sampling results, before-dredge survey data, and CAD drawings (engineer's lines and grades) received from the owner, SES will program the dredging software with the vertical and horizontal extents of all dredging areas and breakouts of material (Landfill, Lot 24, Beneficial Reuse).

2.1 DREDGING SCHEDULE

Figure 1 depicts the tasks, dredge to be utilized and duration of each task. SES's primary objective is to open the beneficial reuse area to allow simultaneous work of both dredges. In order to mechanically dredge and offload the Lot 24 and Landfill material presently in the reuse area, SES may have to first dredge access to the proposed offloading site. Task 1 will be subject to before dredging survey conditions and the measured water elevation at the start of dredging.

Task #	Dredging Task Description	Dredge	Production CY/Day	Work Schedule	Calendar Days
1	Access to Offloading Site	Cat 345	800	6d 12hr	2
2	Dredging of Beneficial Reuse Area	Cat 345	1,600	6d 24hr	20
3a	Dredge & Place Beneficial Reuse (No Overburden Area)	Moray	500	6d 12hr	20
3b	Removal of Landfill Overburden	Cat 345	1,600	6d 24hr	4
4a	Dredge & Place Beneficial Reuse (Overburden Area)	Moray	500	6d 12hr	16
4b	Complete Landfill Material	Cat 345	1,600	6d 24hr	8
5	Complete Lot 24 Material	Cat 345	1,600	6d 24hr	18

Figure 1 Dredging Tasks & Durations

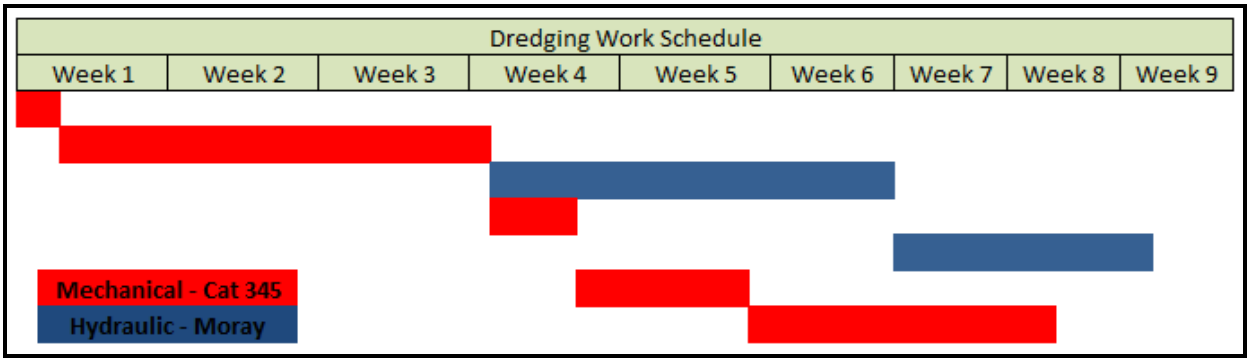


Figure 2 Dredging Schedule Bar Chart

Task numbers refer to dredging tasks only and are provided as a guide to the plan view layout of the dredging plan. Figure 2 presents SES planned schedule for dredging work activities. The red bars represent mechanical dredging activities (Landfill and Lot 24) and the blue bars represent the hydraulic dredging activities (Beneficial Reuse).

2.2 DREDGING PLAN VIEW LAYOUT/FIGURE

The following seven figures are provided as general guidance as to the dredging plan. The plan will be further refined upon receipt of data yet collected and modified as needed throughout the project to adapt to unforeseen issues and or conditions.

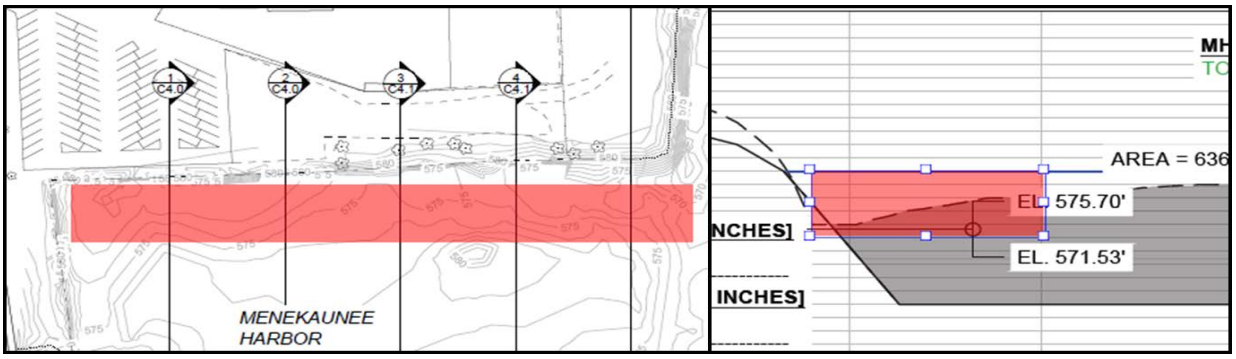


Figure 3 Dredging Task 1

Task 1 includes a minimal amount of mechanical dredging, that may be required to provide access to the planned unlading operation on the dewatering plan. This work as Task 1 will be contingent upon the resutls of the before dredging survey and the site water elevation.

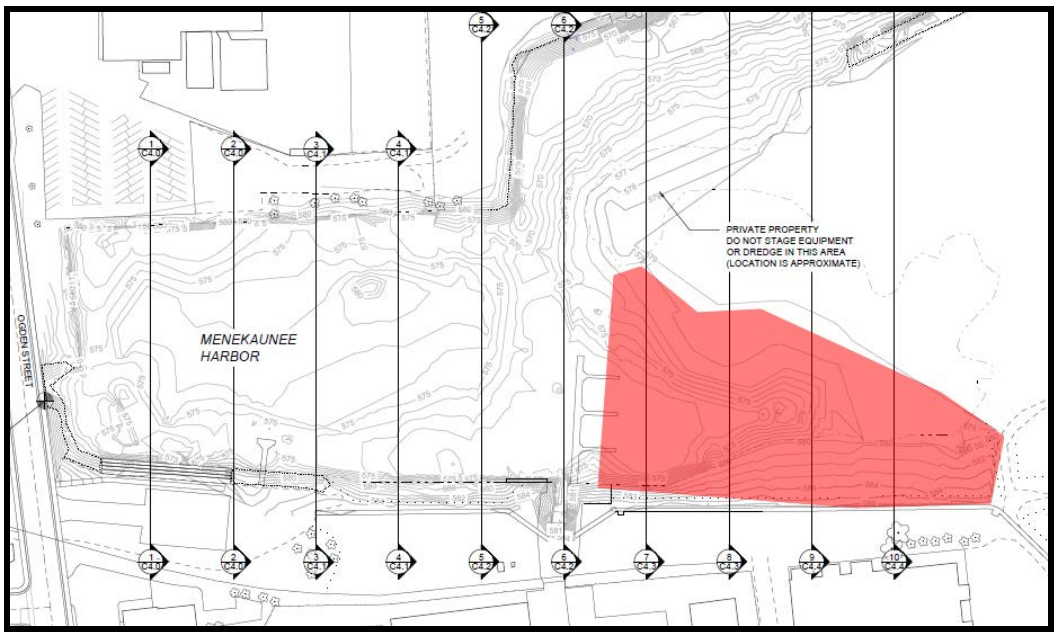


Figure 4 Dredging Task 2

Task 2 includes both landfill and lot 24 dredging that will be dredged seperately and segregated on the dewatering pad. The dredging of this area will be performed by the mechanical excavator.

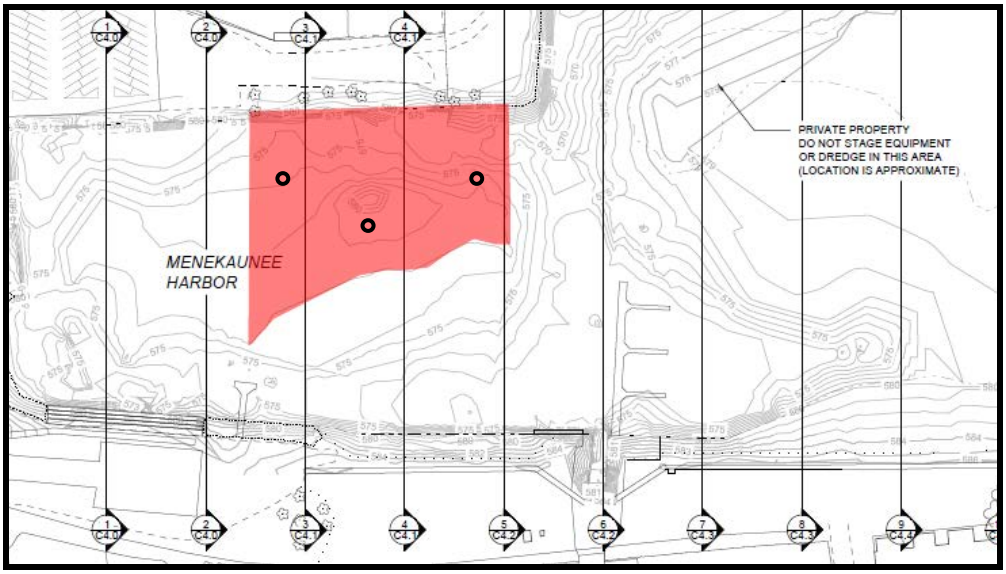


Figure 5 Dredging Task 3a

Task 3a is the hydraulic dredging of the beneficial reuse material with direct placement into the placement area. This region does not include any overburden of Lot 24 or Landfill material. Proposed in-situ sampling locations are identified by the black dots.

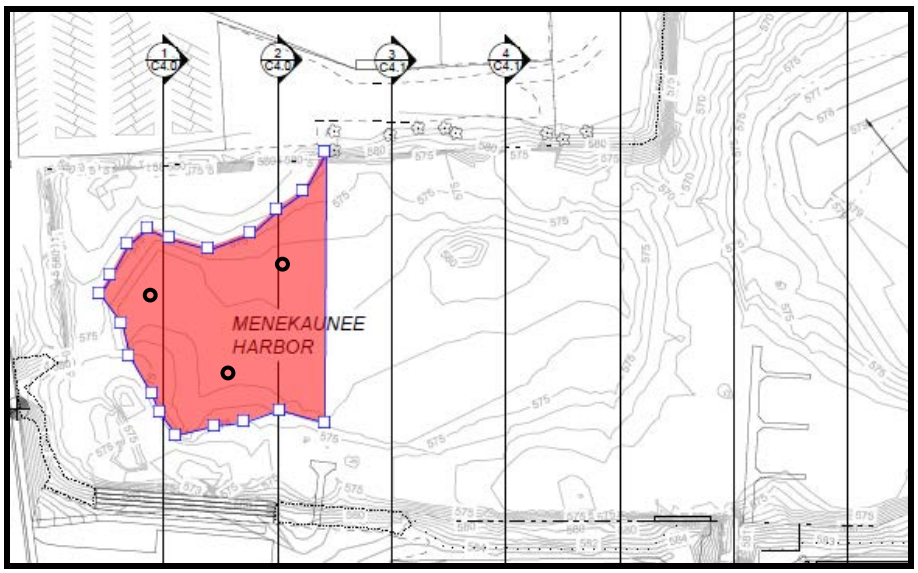


Figure 6 Dredging Task 3b

Task 3b requires mechanical dredging of overburden landfill material. After completion, this area will provide the remainder of the beneficial reuse fill. Proposed in-situ sampling locations are identified by the black dots (we will perform these through the landfill material to verify underlying material is suitable).

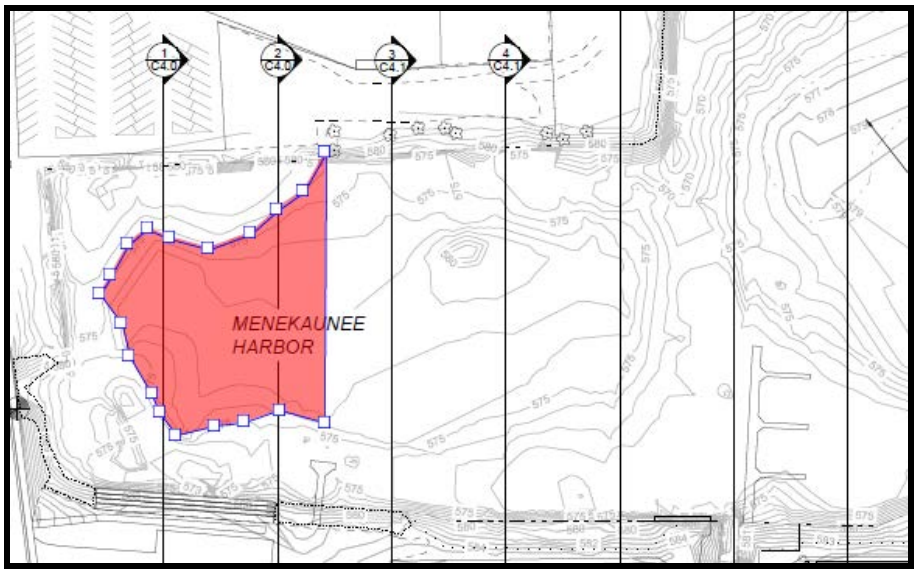


Figure 7 Dredging Task 4a

Task 4a is the hydraulic dredging of the beneficial reuse material with direct pump to the placement area. Material will be dredged hydraulically until the placement area is completed, remaining material will be dredged mechanically for Lot 24 disposal.

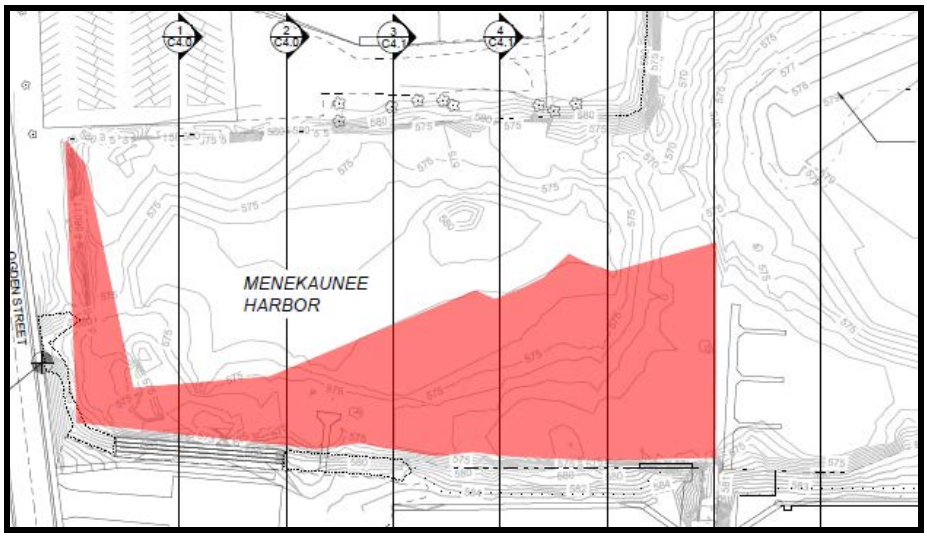


Figure 8 Dredging Task 4b

Task 4b is the mechanical dredging of the remaining Landfill material.

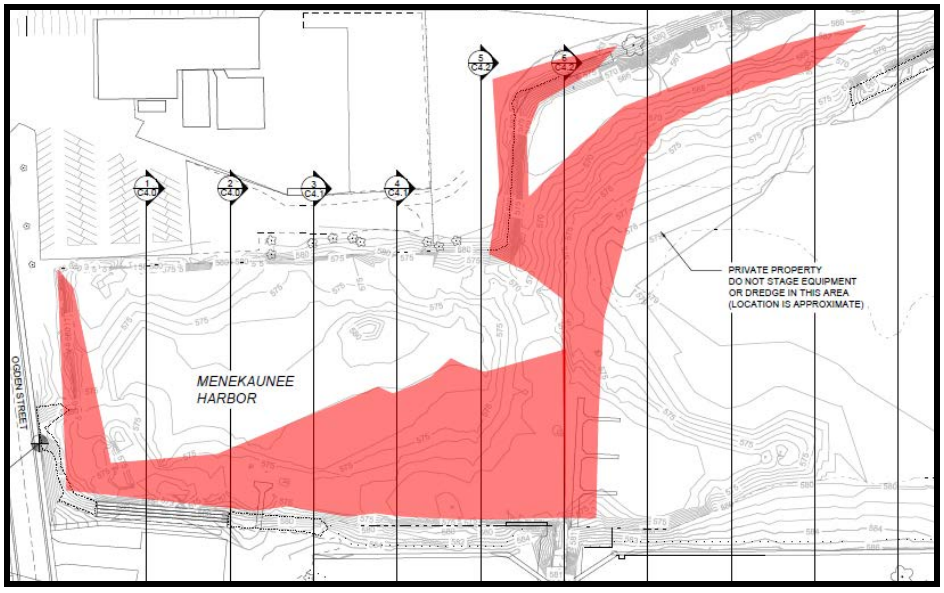


Figure 9 Dredging Task 5

Task 5 is the mechanical dredging of the remaining Lot 24 material.

APPENDIX C

Technical Specifications (abbreviated)

SECTION 03 30 00

CAST-IN-PLACE CONCRETE

PART 1 GENERAL

1.01 SUMMARY

A. Provide cast-in-place concrete work as shown and as specified. Comply with applicable provisions of Divisions 00 and 01.

1.02 SUBMITTALS

A. Shop Drawings: Submit shop drawings for fabrication, bending, and placement of concrete reinforcement. Comply with ACI 315.

B. Product Data: Submit product data for concrete accessories.

C. Mix Designs: Submit proposed mix designs to A/E ten days prior to beginning concrete work. Do not begin concrete production until mixes have been reviewed.

D. Delivery Tickets: Submit delivery ticket to A/E for each load of concrete delivered to project.

E. Test Reports: Testing agency shall submit copy of field and laboratory reports to A/E.

F. Make submittals in accordance with Section 01 01 00.

1.03 QUALITY ASSURANCE

A. Prior to placing concrete, request review of reinforcement steel by A/E.

B. Give A/E two working days notification of all planned concrete pours so that appropriate construction observation can be present at the project site.

1.04 CODES AND STANDARDS

A. Comply with the following codes and standards, except as otherwise designated:

1. ACI 301 Specifications for Structural Concrete.
2. ACI 304 Guide for Measuring, Mixing, Transporting and Placing Concrete.
3. ACI 318 Building Code Requirements for Structural Concrete.
4. ACI SP-2 Manual of Concrete Inspection.

1.05 TESTING

A. Contractor shall arrange and pay for services of a qualified testing agency acceptable to Owner and independent of Contractor.

B. Testing agency shall test concrete to measure slump, entrained-air content, temperature, and compressive strength to determine compliance with specifications. Furnish test apparatus and cylinders, perform on-site sampling and testing, submit samples, and perform laboratory tests.

C. On-site tests shall be performed under observation of A/E unless waived.

D. Perform slump, air content, and temperature tests prior to concrete placement each day, whenever there is a change in consistency of concrete, and when concrete cylinders are prepared. If measured slump, air content, or temperature falls outside specified limits,

immediately check another portion of same batch. In event of a second failure, concrete shall be rejected.

E. During progress of work, prepare three test cylinders per 50 cu yd or fraction thereof for each class of concrete placed each day. Identify samples, moist cure in accordance with ASTM C31, and ship samples to testing laboratory for one 7-day compressive strength test and two 28-day tests.

F. Test procedures shall be in accordance with ASTM C31, C39, C143, C172, C231, and C1064.

G. Cost of tests, including materials and transportation, shall be paid by Contractor and shall be considered incidental to the various items of concrete work.

PART 2 PRODUCTS

2.01 CEMENTITIOUS MATERIAL

A. Cement: Portland cement, ASTM C150, Type I.

B. Fly Ash: ASTM C618, Class C.

2.02 AGGREGATES

A. Fine and coarse aggregates, ASTM C33, consisting of clean, hard, durable sand and crushed rock, crushed gravel, or gravel. Coarse aggregate shall meet grading requirements for size number 67, 57, or 467. Maximum coarse aggregate size for each class of concrete shall be as indicated in Part 4 Schedules. Ratio of coarse aggregate to fine aggregate shall not be less than 1:1 nor more than 2:1.

2.03 WATER

A. Mixing water shall be potable, free of oil, acid, excessive alkalinity, organic matter, and salts.

2.04 ADMIXTURES

A. Air-entrained admixture shall conform to ASTM C260. Other admixtures which do not adversely affect strength and durability of concrete may be used with permission of A/E, if used in strict accordance with manufacturer's instructions. Care shall be exercised to assure that the admixture does not increase or decrease air content outside of allowable limits. Do not use salt or chemical anti-freeze admixtures.

2.05 REINFORCING STEEL

A. ASTM A615, Grade 60, new billet steel bars.

B. Provide metal chairs, stirrups, spacers, and hangers to support reinforcement and insure against displacement during placement of concrete. For exposed-to-view concrete surfaces, where legs of supports are in contact with forms, provide supports with legs which are plastic protected (CRSI, Class 1) or stainless steel protected (CRSI, Class 2).

2.06 FORMWORK

A. Forms for Exposed Finish Concrete: Construct forms for exposed concrete surfaces with water-resistant plywood, metal, metal-framed plywood-faced, or other acceptable panel type materials, to provide continuous, straight, smooth, exposed surfaces. Furnish in largest practicable sizes to minimize number of joints and to conform to joint system if shown. Provide form material with sufficient thickness to withstand pressure of newly placed concrete without bow or deflection. Rust-stained steel forms are not acceptable.

B. Forms for Unexposed Finish Concrete: Construct forms for concrete surfaces which will be unexposed in finished structure with plywood, lumber, metal, or other acceptable material. Provide lumber that is dressed on at least one side and two edges for tight fit.

2.07 CURING COMPOUND

A. White, waterborne, membrane-forming curing compound, ASTM C309, Type 2, Class B, dissipating.

2.08 CONCRETE MIXTURES

A. Conform to minimum standards for class and usage in Part 4 Schedules.

B. Prepare design mixes for each type of concrete on the basis of compressive strength by methods recommended in ACI 318. Use an independent materials laboratory for preparing and reporting proposed mix designs.

PART 3 EXECUTION

3.01 PREPARATION

A. Wet forms with form release agent.

B. Sprinkle subgrade with water.

C. Separate old concrete and adjacent structures from new pour with expansion joint filler.

D. Provide concrete cover over reinforcing steel as designated on the Drawings.

3.02 JOINTS

A. General: Construct joints true to line with faces perpendicular to surface plane of concrete.

B. Construction Joints: Locate and install construction joints so that strength and appearance of concrete is not impaired, at locations shown or as approved by A/E.

C. Contraction Joints in Walls: Provide contraction (control) joints in walls as shown. If not shown, provide a contraction joint within 10 to 15 ft of wall corner and at a maximum spacing of 25 ft thereafter.

1. Total depth of inside and outside contraction joints shall be 1/4 of wall thickness.

3.03 MIXING CONCRETE

A. "Ready-mix" concrete shall be produced, delivered and handled in accordance with ASTM C94. Concrete shall be deposited at job site within one hour after introduction of water in mix. Care shall be taken in transferring concrete from truck or mixer to avoid segregation of aggregates in mixture.

3.04 CONCRETE PLACEMENT, GENERAL

A. Place concrete as specified and in accordance with ACI 304. Maintain reinforcing steel in proper position.

B. Deposit concrete continuously or in layers of such thickness that no concrete will be placed on concrete which has hardened sufficiently to cause formation of seams or planes of weakness. If a section cannot be placed continuously, provide construction joints. Deposit concrete as near as practicable to final location to avoid segregation from rehandling or flowing. Do not subject concrete to any procedure which will cause segregation.

C. Screed concrete to proper level to avoid excessive skimming or grouting.

D. Do not use concrete which becomes non-plastic and unworkable, or does not meet required quality control limits, or which has been contaminated by foreign materials. Do not use retempered concrete. Remove rejected concrete from project site.

E. Rock pockets or voids found after forms are removed shall be filled immediately with a well-mixed grout composed of one part of Portland cement and three parts of fine aggregate finished to true surface of concrete.

F. Concrete shall not be placed around castings, frames, joints, and other embedded items until they have been accurately adjusted and set to required alignment and grade. Prior to placing of concrete, castings, frames, and embedded metal fixtures shall be painted on their contact surface with a heavy coat of asphaltic mastic or separated with expansion joint filler.

3.05 PLACING CONCRETE INTO FORMS

A. Deposit concrete in forms in horizontal layers not deeper than 18 in. and in a manner to avoid inclined construction joints.

B. Remove temporary spreaders in forms when concrete placing reaches elevation of spreaders.

C. Consolidate concrete in forms by mechanical vibrating equipment and supplement by hand-spading, rodding or tamping. Use vibrators designed to operate at a speed of not less than 6000 impulses per minute when submerged in concrete. Vibration of forms and reinforcing will not be permitted.

D. Do not use vibrators to move concrete inside forms. Insert and withdraw vibrators vertically at uniformly spaced locations not farther than visibly effective. Do not insert vibrators into lower layers of concrete that have begun to set. At each insertion, limit duration of vibration to time necessary to consolidate concrete and complete embedment of reinforcement and other items without segregation of mix.

3.06 COLD WEATHER PLACING

A. Protect concrete work from physical damage or reduced strength caused by frost, freezing actions, or low temperatures, in compliance with ACI 306 and as specified below.

1. When air temperature falls to or is expected to fall below 40 deg F, uniformly heat water and aggregates before mixing to obtain a concrete mixture temperature of not less than 60 deg F (50 deg F for heavy sections) and not more than 90 deg F at point of delivery.
2. Do not use frozen materials or materials containing ice or snow. Do not place concrete on frozen subgrade or on subgrade containing frozen materials. Ascertain that forms, reinforcing steel, and adjacent concrete surfaces are entirely free of frost, snow and ice before placing concrete.
3. During seasons when atmospheric temperature may be expected to drop below 40 deg F, concrete shall be protected by covering with impermeable paper and not less than 12 in. of loose dry hay or straw. Retain covering for ten days.

3.07 HOT WEATHER PLACING

A. When hot weather conditions exist that would seriously impair quality and strength of concrete, place concrete in compliance with ACI 305 and as specified below.

1. Cool ingredients before mixing to maintain concrete temperature at time of placement below 80 deg F. Mixing water may be chilled, or chopped ice may be used to control temperature provided water equivalent of ice is calculated in total amount of mixing water.

2. Cover reinforcing steel with water-soaked burlap if it becomes too hot, so that steel temperature will not exceed the ambient air temperature immediately before embedment in concrete.
3. Spray forms, reinforcing steel, and subgrade just before concrete is placed.
4. Do not use set-control admixtures, unless approved by A/E.

3.08 FINISHING FORMED SURFACES

A. Provide standard rough finish to formed surfaces to be concealed in finish work or by other construction, unless otherwise designated. Standard rough form finish shall be the concrete surface having texture imparted by form facing material, with defective areas repaired and patched and fins and other projections exceeding 1/4 in. in height rubbed down with wood blocks.

B. Provide standard smooth finish for formed surfaces exposed-to-view or to receive a covering applied directly or bonded to concrete, such as waterproofing, dampproofing, or paint. Standard smooth finish shall be the as-cast concrete surface obtained with form facing material, with defective areas repaired and patched and fins and other projections on surface completely removed and smoothed.

3.09 FINISHING UNFORMED SURFACES

A. Thoroughly float surface after concrete has been struck off. Check and level surface plane to a tolerance not exceeding 1/4 in. in 10 ft when tested with a 10 ft straightedge placed on surface at not less than two different angles.

B. Exterior surfaces shall have a lightly broomed surface with grain perpendicular to direction of travel.

C. Edges shall be neatly trimmed with 1/4 in. radius edging tool.

3.10 CURING AND SEALING

A. General: Protect freshly placed concrete from premature drying and excessive cold or hot temperatures. Cure formed and unformed concrete for seven days or until 75 percent of the required 28-day compressive strength is obtained, whichever is less.

B. Curing Compound: Apply white-pigmented membrane curing compound to exposed surfaces, unless otherwise indicated.

3.11 CONCRETE SURFACE REPAIRS

A. Formed Surfaces: Request inspection of concrete surfaces immediately after removal of formwork. After inspection, repair and patch defective areas as soon as practicable.

1. Remove bulges and projections by chipping or tooling. Rub or grind surface after removal.
2. For patching, use dry-pack mortar consisting of one part portland cement to 2-1/2 parts fine aggregate passing a No. 16 mesh sieve and only enough water as required for handling and placing. Cut out honeycomb, rock pockets, voids over 1/4 in. diameter, and holes left by tie rods and bolts. Remove defects down to solid concrete but, in no case, to a depth of less than 1 in. Make edges of cuts perpendicular to concrete surface or slightly undercut to provide a key at edge of cut. Thoroughly clean, dampen with water, and brush-coat area to be patched with bonding agent. Place patching mortar before bonding agent has dried. Compact mortar in place and strike off slightly higher than surrounding surface. For exposed-to-view surfaces, patch shall match color of surrounding concrete.
3. For patching less than 1 in. in depth, Contractor may propose a proprietary patching product in lieu of removing concrete to minimum depth of 1 in. Submit proposed product to A/E for approval.

B. Unformed Surfaces: Repair surfaces that do not meet specified requirements.

1. Correct low and high areas. Submit proposed repair products to A/E for approval.
2. Fill cracks with an approved epoxy mortar which will match slab finish in density and performance. Grind filler smooth and even with adjacent surface, free of bumps or depressions at joint.

PART 4 SCHEDULES

4.01 CLASSES OF CONCRETE

Class	Min. Comp. Strength @ 28 days, p.s.i.	Max. Slump	Max. Agg. Size	Min. Cement, Bags/ C.Y.*	Max. Water-Cement Ratio	Air Content, % By Volume
AA	4000	4"	3/4"	6	0.45	5-8%

* Fly ash shall be used to constitute between 15 to 30% by weight of total cementitious material.

4.02 USAGE SCHEDULE

A. Use class AA concrete for all work.

END OF SECTION

SECTION 05 50 00

METAL FABRICATIONS

PART 1 GENERAL

1.01 SUMMARY

A. Provide metal fabrications as shown and as specified. Comply with applicable provisions of Divisions 00 and 01.

1.02 RELATED SECTIONS

03 30 00 Cast-In-Place Concrete.
31 41 22 Steel Sheet Piling.

1.03 COORDINATION

A. Furnish inserts and anchoring devices to be embedded in concrete for installation of miscellaneous metal work. Provide setting drawings, templates, and instructions for installation of anchorage devices. Coordinate delivery with related work to avoid delays.

1.04 SUBMITTALS

A. Shop Drawings: Submit shop drawings for fabrication and erection of miscellaneous metal assemblies. Include product data, load tables, layouts, elevations, details of sections, connections, anchorage and accessory items. Provide templates for anchors and bolts installed under other sections.

B. Make submittals in accordance with Section 01 01 00.

1.05 FIELD MEASUREMENTS

A. Take measurements prior to shop fabrication. Allow for trimming and fitting to make field adjustments. Correct defects resulting from failure to take proper measurements.

PART 2 PRODUCTS

2.01 STEEL

A. Shapes, Plates, and Bars: ASTM A572, Grade 50.

B. Anchor Rod: ASTM A615, Grade 75, continuously threaded; Dywidag Threadbar by Dywidag-Systems International or Williams Form Engineering Corporation All-Thread Rebar.

C. Pipe: ASTM A53, standard weight (Sch. 40) unless otherwise indicated.

2.02 FASTENERS

A. High-strength bolts, nuts and washers shall conform to ASTM A325, Type 1. All components shall be galvanized to comply with ASTM A153; except fasteners 1/2 in. or less in diameter may be coated with electrodeposited zinc or cadmium, ASTM B633.

B. Nuts and washers shall be furnished by anchor rod manufacturer.

2.03 GALVANIZING REPAIR PAINT

A. High zinc-dust content paint for regalvanizing welds in galvanized steel, complying with SSPC-Paint 20.

2.04 FABRICATION

A. Use materials of designated type, size, and thickness or, if not shown, of required strength, stiffness, and durability. Work to field measurements and shop drawings, using proven details of fabrication and support. Miscellaneous framing and support members shall comply with AISC Specification.

B. Where exposed to view, use materials that are smooth and free of surface blemishes such as pitting, seam marks, roller marks, rolled trade names, and roughness.

C. Form work true to line and level with accurate angles and surfaces and straight sharp edges. Ease exposed edges to a radius of approximately 1/32 in. unless otherwise shown. Form bent-metal corners to smallest radius possible without causing grain separation or other impairment. Shearings and punchings shall be clean and true.

D. Welding of anchor rods is not permitted.

E. Provide anchorage devices and fasteners for securing miscellaneous metal items to in-place construction. Cut, reinforce, drill, and tap miscellaneous metal work as required to receive hardware and connections required by other trades.

F. Preassemble and fit items to minimize field splicing and assembly. Disassemble units only as necessary for shipping and handling limitations. Clearly identify units for reassembly and installation.

2.05 GALVANIZING

A. Galvanize all metal fabrications (except sheet piling and sheet pile cap) in accordance with ASTM A123 for iron and steel products.

B. Galvanize after fabrication where practicable.

PART 3 EXECUTION

3.01 INSTALLATION

A. Install anchorage devices and fasteners for adequate support. Perform cutting, drilling, and fitting, as required. Set work accurately in location, alignment, and elevation, plumb, level, true, and free of rack, measured from established lines. Provide temporary bracing or anchors in formwork for items to be built into concrete, masonry, or similar construction.

B. Fit exposed connections to form tight hairline joints. Field weld connections which cannot be shop welded because of shipping size limitations. Comply with AWS Code for manual shielded metal-arc welding, appearance and quality of welds, and methods for correcting welding work. Grind exposed joints smooth and touch-up with shop primer. Do not weld, cut, or abrade surfaces of exterior units which have been hot-dip galvanized after fabrication and are intended for bolted or screwed field connections.

C. Remove slack in anchor rods.

3.02 ANCHOR ROD TENSIONING

A. Anchor Rod Testing Equipment:

1. Testing equipment shall consist of a hydraulic jack with calibrated pressure gage for applying the load and a dial gage or vernier scale to measure anchor movement.
2. Stressing equipment shall have a capacity sufficient to stress anchors to the specified required loads within the rated capacity in one stroke.
3. Pressure gage shall be graduated in 50 psi increments.
4. Pumps shall be capable of applying each load increment in less than 60 seconds and shall be capable of maintaining hydraulic pressure within 50 psi.
5. Equipment shall permit stressing of anchor in increments and raising or lowering the load in anchor.
6. Equipment shall be calibrated with an accuracy of +/- 2% and the calibration certificate and graphs shall be available at site.
7. Stressing equipment and pressure gage shall have been calibrated as a unit no more than 30 calendar days prior to commencing work.

B. Anchor Rod Stressing:

1. Prior to stressing, surfaces upon which stressing equipment is resting shall be smooth and clean and stressing equipment shall be aligned as nearly with center of hole as possible.
2. Post-tension and lock-off load shall be 7.00 kips.
3. Contractor shall determine lock-off procedure so that lift-off results meet the acceptance criteria specified. Note several passes of stressing may be required before required load can be maintained.
4. Maximum stress shall never exceed 8.00 kips.
5. Safety precautions shall be taken to prevent workers from being behind or above stressing equipment during stressing.

C. Anchor Rod Final Lock-Off:

1. A lift off test shall be made only after all anchors have been stressed permanently to verify the load in each anchor before anchor is permanently locked-off.
2. Lift-off reading shall be within 5% of the specified lock-off load. If lift-off reading is not within 5% of the specified lock-off load, anchorage shall be reset and another lift-off reading shall be obtained.

3.03 TOUCH-UP

A. Immediately after erection, clean fasteners and abraded areas. Repair damage to galvanized surfaces in accordance with ASTM A780.

END OF SECTION

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SECTION 31 05 10

SITE PREPARATION

PART 1 GENERAL

1.01 SUMMARY

A. Provide site preparation as shown and as specified. Comply with applicable provisions of Divisions 00 and 01.

B. Work includes, but is not limited to:

1. Protection of improvements, plants, and utilities.
2. Removal and replacement of improvements.
3. Location of utilities and coordination with utility companies.
4. Clearing and grubbing trees and vegetation.
5. Topsoil salvage.
6. Site demolition and abandonments.

PART 2 (NOT USED)

PART 3 EXECUTION

3.01 PROTECTION

A. Protect improvements on site and on adjoining properties. Provide barricades, coverings, or other types of protection as necessary to prevent damage and to safeguard against injury. Restore to original condition improvements damaged by the work or improvements which required temporary removal during construction.

B. Protect existing vegetation indicated to remain against unnecessary cutting, breaking, bruising, or smothering by stockpiling excavated materials or parking of vehicles within drip line. Provide temporary fences, tree wells, barricades, or guards; repair or replace trees and vegetation damaged by construction operations.

C. Maintain survey monuments, reference points, and benchmarks; notify Owner and A/E of disturbance to markers.

D. No extra payment or time will be allowed for protection work that could have been suspected or anticipated by site inspection and interpretation of bidding documents prior to execution of contract.

3.02 LOCATING EXISTING UTILITIES

A. Location and description of underground utilities and structures shown on drawings are approximate and are based on records available to Owner and A/E, or surface features indicating their existence. There may be other utilities within project area that are not shown.

B. Notify all affected utility companies of construction operations at least three working days before beginning work near their facilities. Do not begin excavation work until underground utility locations have been marked.

C. Use caution when excavating so that exact location of underground utilities, both known and unknown, may be determined. Provide adequate protection and support for utilities during construction operations.

D. If uncharted or incorrectly charted utilities are encountered during excavation work, or if proposed construction conflicts with existing utilities, give prompt notice and submit proposed solution to A/E for approval. Cooperate with Owner, and public and private utility companies, to keep their services and facilities in operation. Repair damaged utilities to satisfaction of utility owner.

3.03 SITE CLEARING AND GRUBBING

A. Remove trees, stumps, snags, shrubs, brush, heavy growths of grass, weeds and other vegetation, improvements, rubbish and debris, and obstructions that interfere with proposed construction; remove items only as necessary for completion of work.

1. At the Lot 24 Containment Site, clearing and grubbing shall be completed as necessary to establish the Containment Site area and its surrounding storm water drainage swale; the storm water drainageway traveling between the Containment Site area and the existing detention basin; and, the project's topsoil stockpile,

B. Cut brush and vegetation flush with ground. Grub out stumps, roots having a diameter of 2 in. or larger, and root clusters to a depth of at least 2 ft below subgrade elevation for pavements, structures, and embankments and 6 in. below ground surface in other areas.

C. Carefully and cleanly cut roots and branches of trees indicated to be left standing, where such roots and branches obstruct new construction. Cut back roots a minimum of 1 ft from concrete work, paving, and structures and to a depth of not less than 2 ft below structures, foundations, and embankments.

D. Branches and removed roots shall be ground up on site and thinly spread on top of Containment Site area base grade. Logs from trees greater than 3 in. in diameter shall be set aside and piled for later loading into Owner-provided vehicle for Owner's use.

3.04 TOPSOIL STRIPPING AND STOCKPILING

A. Topsoil shall include all friable, fertile, loam soil suitable for grass and plants, found at surface, reasonably free of subsoil, clay lumps, stones, objects over 2-in. diameter, weeds, large roots, root clusters, and other objectionable material. Objectionable material (not suitable for stockpiling and reuse during restoration) shall be piled within Lot 24 Containment Site for disposal.

B. Strip topsoil from project area to whatever depths encountered; prevent intermingling with underlaying subsoil or other objectionable material. Remove heavy growths of grass from areas before stripping topsoil. Objectionable turf (not suitable for stockpiling and reuse during restoration) shall be piled within Containment Site area for disposal.

C. Where trees are indicated to remain (at edge of limits of construction), terminate stripping a sufficient distance from such trees to prevent damage to root system.

D. Stockpile topsoil in storage piles in areas where designated on Drawings. Construct storage piles to freely drain surface water. Control windblown dust as appropriate in accordance with WDNR requirements and project plans.

3.05 DEMOLITION (HARBOR AREA)

A. Remove timber pile walls and other improvements within construction limits as shown and as required for construction.

B. Cut off existing timber pile wall to be removed a minimum of 2 ft below the structural fill or riprap bedding. In locations where new sheet pile wall is constructed, remove existing timber to completely behind new sheeting and 2 ft below final grade on harbor side.

C. Owner shall have first right to retain all useful salvage. All items not retained by Owner and construction debris shall become property of Contractor.

3.06 DEBRIS DISPOSAL (HARBOR AREA)

A. Remove debris and excess materials from site and legally dispose of it; do not burn debris.

END OF SECTION

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SECTION 31 22 00

GRADING

PART 1 GENERAL

1.01 SUMMARY

A. Provide grading as shown and as specified. Comply with applicable provisions of Divisions 00 and 01.

B. This section does not apply to earthwork associated with dredging operations. Refer to Section 35 20 13 for dredging.

1.02 RELATED SECTIONS

31 05 10 Site Preparation.
32 15 20 Crushed Aggregate Surfacing.

1.03 CLASSIFICATION

A. Excavation of materials encountered under this work will be unclassified without regard to type, difficulty to remove, or suitability for use in construction.

1.04 SUBMITTALS

A. Test Reports:

1. Two weeks prior to start of construction, indicate source and submit gradation analysis of proposed fill and backfill materials.
2. Submit reports for laboratory and field tests required under "Testing" article. Test reports for slab and pavement subgrades shall be submitted prior to placing concrete or paving materials.

B. Make submittals in accordance with Section 01 01 00.

1.05 TESTING

A. Contractor shall arrange and pay for soil sampling and testing by a qualified testing agency, acceptable to Owner and independent of Contractor. Test soil materials for suitability for intended purpose.

B. Test subgrade and fill materials for gradation in accordance with ASTM C136 for conformance with ASTM D2487 gradation limits. Test materials for liquid limit and plasticity index in accordance with ASTM D4318.

C. Provide one optimum moisture-maximum density curve for each type of soil encountered in subgrade and fills under structure slabs and foundations; determine maximum densities in accordance with ASTM D1557.

D. During course of work, testing agency shall inspect and approve subgrades and fill layers before further construction work is performed on each layer. Perform field density tests in accordance with standard, recognized procedures. Take tests as follows:

1. Sheet Pile Wall Areas: Perform at least one field density test on fill subgrade for every 10,000 sq ft of new fill area, but in no case less than three tests. In each compacted fill layer, perform one field density test for every 10,000 sq ft of new fill area, but in no case less than three tests.
2. Lot 24 Access Road: No compaction testing required.

E. If in opinion of A/E, based on reports of testing agency and inspection, subgrade or fills which have been placed are below specified density, provide additional compaction and testing at no additional cost to Owner.

1.06 PROTECTION

A. Protect existing improvements, utilities, trees and shrubs, and reference marks in accordance with Section 31 05 10.

PART 2 PRODUCTS

2.01 SOIL MATERIALS, GENERAL

A. Soil materials shall be free of organic matter, debris, frozen soils, ice, and other objectionable materials. Rock particles larger than maximum size specified shall be removed prior to placement of soil.

B. Select existing material from required excavations may be used for fill or backfill if it meets the specified product requirements. If necessary, furnish additional approved material from suitable off-site sources.

2.02 STRUCTURAL FILL

A. Select soils consisting of a predominantly sand material, or a gravel base course, with 100% passing the 1-in. sieve, 70-100% passing the #4 sieve, and less than 15% passing the #200 sieve.

2.03 GENERAL SITE FILL

A. Select, natural, free draining soils complying with ASTM D2487 soil classification groups GW, GP, SW, SP, GM, GC, SM, SC, or combinations thereof, and suitable for compaction. Maximum aggregate size shall be 1/2 specified lift thickness.

2.04 AGGREGATE FILL

A. Aggregate fill for access road construction shall be in accordance with Section 32 15 20.

PART 3 EXECUTION

3.01 GRADING

A. Grade areas within project limits to achieve cross sections, lines, and elevations indicated. Finish surface to be reasonably smooth and free from irregular surface changes. Provide a smooth transition between adjacent existing grades and new grades.

B. For turf areas, allow 6 in. for topsoil placement. For surfaced areas such as slabs, pavements, and walks, grade to underside of respective surfacing or base course.

C. Finish subgrades to required elevations within the following tolerances:

1. Lawn or Unpaved Areas: Plus or minus 1 in.
2. Lot 24 Access Road: Plus or minus 1 in.

3.02 SUBGRADE EXAMINATION AND PREPARATION

A. Examine subgrade prior to placement of fill. Remove organic materials and debris subject to rot or corrosion. Plow, strip, or break-up sloped surfaces steeper than 1 vertical to 4 horizontal so that fill material will bond with subgrade.

B. In sheet pile wall areas, proof-roll exposed subgrade with a large vibratory roller rated for a minimum compactive force of 20,000 lb to compact subgrade and detect areas which must be undercut or improved. Inform A/E of unsuitable, unconsolidated subgrade soils.

C. After subgrade soil is stable, scarify top 6 to 8 in., moisture condition, and compact surface to density specified in Part 4 Schedules.

D. Reconstruct subgrades damages by freezing temperatures, frost, rain, accumulated water, or construction activities, as directed by A/E, without additional compensation.

3.03 ADDITIONAL EXCAVATION (OVER EXCAVATION)

A. If unsuitable bearing materials, such as poorly compacted fill, existing foundations, rubble, debris, or organic deposits, are encountered at required subgrade elevations, carry excavations deeper and replace excavated material with properly compacted Structural Fill as directed by A/E.

B. Where over excavation below footing subgrade is required, widen over excavation beyond footing edges at least 1 ft for each 1 ft of over excavation depth.

C. Removal of unsuitable material and its replacement as directed will be paid for as extra work, unless a pay item is included in the Bid Schedule. Do not proceed with extra or unit price work until authorized.

3.04 FILLING

A. Do not place fill until subgrade preparation has been examined and approved by testing agency.

B. Place and compact fill materials in layers to required elevations as follows:

1. Under grass and planted areas: Use General Site Fill.
2. Around sheet pile wall and deadman: Use Structural Fill.
3. For Lot 24 access road: Use aggregate fill.

C. Place fill in approximately horizontal layers; do not exceed maximum lift thickness specified in Part 4 Schedules before compaction.

D. During placement and compaction, maintain moisture content of materials within optimum range. Compact each layer of fill to not less than the percentage of maximum density specified in Part 4 Schedules.

E. Do not place fill on frozen subgrade.

3.05 CONTAINMENT SITE PERIMETER DRAINAGE SWALE

A. The perimeter drainage swale shall be minimally sloped to direct water around the site perimeter to the drainageway to direct storm water to the existing detention basin. Contractor shall attempt to construct and grade drainage swale to the extent possible without impacting sandy soils located beneath existing site topsoil layer.

B. Following completion of drainage swale, the swale and disturbed areas (outside the limits of the Containment Site area) shall be immediately seeded in accordance with Section 32 92 00. Erosion control devices shall be immediately installed in accordance with Section 01 57 19.

C. Surveying of drainage swale and affected areas will be performed by A/E to confirm positive drainage and acceptable contour.

3.06 MAINTENANCE

A. Protect newly graded areas from traffic and erosion, and keep free of trash and debris. Repair and re-establish grades in settled, eroded, and rutted areas to specified tolerances.

B. Maintain erosion control measures to prevent run-off and sediment pollution of adjacent water courses.

3.07 DISPOSAL OF EXCESS AND WASTE MATERIALS

A. Remove excess excavated material, trash, debris, and other waste materials and legally dispose of them off-site.

PART 4 SCHEDULES

4.01 COMPACTION SCHEDULE

Location	Lift Thickness	Compaction [1]
Sheet Pile Wall Areas	8"	95%
Lot 24 Access Road	8"	90%
Unpaved Areas	12"	90%

[1] Percent of maximum density determined in accordance with ASTM D1557 (Modified Proctor test).

END OF SECTION

SECTION 31 37 00

RIPRAP

PART 1 GENERAL

1.01 SUMMARY

A. Provide loose rock riprap, including (as indicated on the Drawings) geotextile filter fabric or bedding material, as shown and as specified. Comply with applicable provisions of Divisions 00 and 01.

1.02 SUBMITTALS

A. Product Data:

1. Submit information on sources of riprap and bedding. Provide access to sources to enable A/E to inspect and obtain samples. Do not deliver riprap until reviewed by A/E.
2. Submit fabric product data. Include material samples, certification of physical properties, and installation procedures.

B. Make submittals in accordance with Section 01 01 00.

1.03 TESTING

A. A/E may perform tests to verify that riprap and completed work meet specified requirements. However, these tests are not intended to provide Contractor with information it may need to assure that materials and workmanship meet requirements of specifications, and their performance will not relieve Contractor of responsibility of performing its own tests for that purpose.

PART 2 PRODUCTS

2.01 RIPRAP

A. Durable field or quarry stone that is sound, hard, dense, resistant to the action of air and water, and free of seams, cracks, or other structural defects. Use stone pieces with a length and width no more than twice the thickness.

B. Riprap gradation shall comply with requirements of WIDOT Std. Spec., Section 606, for light riprap (Lot 24) or heavy riprap (Menakaunee Harbor) as indicated on the Drawings.

2.02 GEOTEXTILE FILTER FABRIC

A. Fabric shall be a woven or nonwoven polyester, polypropylene, stabilized nylon, polyethylene, or polyvinylidene chloride material whose function is to pass ground water from beneath fabric while restricting migration of subgrade soil particles into overlying stone ballast. Fabric shall be treated to ensure stability under ultraviolet radiation (sunlight).

B. Fabric shall comply with requirements of WIDOT Std. Spec., Section 645, for Type R fabric.

2.03 BEDDING MATERIAL

A. Riprap bedding materials shall be pervious mixtures of sand and gravel reasonably well-graded from 3/16 to 3 in. in maximum dimensions, but may contain materials less than 3/16 in. in quantities not to exceed the amount required to fill the voids between the materials larger then 3/16 in: *Provided*, that the material shall not contain more than 5 percent, by weight, of material passing the No. 200 sieve.

B. Should gravel, cobbles, or boulders having dimensions of more than 3 in. be found in otherwise approved materials, they shall be removed by Contractor either at the site of excavation or after being placed.

PART 3 EXECUTION

3.01 SUBGRADE PREPARATION

A. Grade subgrade surfaces to lines and grades as shown with an allowance for riprap. Remove organic materials. Compact soft subgrade soils. When fill to achieve subgrade lines is required, provide granular materials.

3.02 FABRIC INSTALLATION

A. Provide fabric under all riprap, unless otherwise shown. Install fabric as shown and in accordance with manufacturer's recommendations.

B. Surface to receive fabric shall be smooth and free of obstructions, depressions, and debris. Lay fabric parallel to direction of water flow.

C. If lapping of fabric is required, minimum overlap shall be 2 ft. Overlaps may be eliminated if fabric sections are either factory or field sewn. Seam strength shall be at least 80% of fabric tensile strength.

D. Secure fabric in place to prevent shifting before or during placement of stone or riprap.

E. Repair or replace torn or punctured fabric in accordance with manufacturer's instructions; no extra compensation will be allowed.

3.03 BEDDING MATERIAL

A. Provide bedding material under all riprap, unless otherwise shown.

B. Surface to receive bedding material shall be smooth and free of obstructions, depressions, and debris.

C. The bedding material need not be compacted in place, but shall be placed in such a manner as will result in uniform layers of bedding for riprap of the specified thickness.

3.04 EQUIPMENT-PLACED ROCK RIPRAP

A. Riprap shall be placed to full course thickness in one operation from base of slope upward; height of riprap freefall shall not exceed 1 ft. Riprap shall be reasonably homogeneous with larger rocks uniformly distributed and firmly in contact and smaller rocks and spalls rammed into voids between larger rocks to interlock and form an even surface.

B. Hand placement will be required where necessary to correct obvious irregularities and to prevent damage to adjacent improvements and wherever equipment placement methods are unsatisfactory.

3.05 HAND-PLACED RIPRAP

A. Riprap shall be securely bedded with larger rocks firmly in contact one to another. Spaces between larger rocks shall be filled with smaller rocks and spalls. Smaller rocks shall not be grouped as a substitute for larger rock. Flat slab rock shall be laid on edge.

END OF SECTION

SECTION 31 37 12

LEDGE STONE

PART 1 GENERAL

1.01 SUMMARY

A. Provide ledge stone as shown and as specified. Comply with applicable provisions of Divisions 00 and 01.

1.02 SUBMITTALS

A. Product Data: Submit information on sources of ledge stone.

B. Samples: Submit samples of stone material indicating colors and finishes available. A/E will choose stone and finish from samples provided.

C. Make submittals in accordance with Section 01 01 00.

1.03 QUALITY ASSURANCE

A. Installer Qualifications: Engage an experienced Installer who has completed installations similar in material, design, and extent to that indicated for this Project and with a record of successful in-service performance.

1.04 DELIVERY AND STORAGE

A. Deliver, store and handle material in a manner to avoid soiling or breaking of stone material.

1.05 PROJECT CONDITIONS

A. Protect adjacent work from damage, soiling and staining during operations.

B. Coordinate the work of this section with adjacent work and coordinate installation of stone materials with the contractor.

PART 2 PRODUCTS

2.01 LEDGE STONE

A. Use indigenous granite and limestone boulders and materials ranging in sizes indicated on the schedule. Natural variations in color and markings, which are characteristics of the stone materials and do not impair strength or appearance, are acceptable. Provide only sound stone, free from defects detrimental to appearance and durability. Provide granite and limestone materials from:

1. Eden Stone Company, Eden, Wisconsin, 920.477.2521.
2. Halquist Stone, Sussex, WI, 800.255.8811, info@halquist.com.
3. Approved Equal.

2.02 GRANULAR GRAVEL BACKFILL

A. Granular gravel backfill- Clean and free of fines, 3/4 to 2 in. range.

2.03 SOIL SEPARATOR FABRIC

A. Soil separator fabric shall be a nonwoven polypropylene, polyethylene, or polyamide material; Propex "Geotex 401", Tencate/Mirafi "140N", Thrace-LINQ "140EX", or approved equal.

PART 3 EXECUTION

3.01 PREPARATION

A. Examination: Examine finished surfaces and grades before commencing work. Do not begin work until unsatisfactory conditions are corrected.

B. Excavation: Excavate to required subgrade depth, allowing space for base preparation and backfill material. Rough grading shall be in place before placing stone materials.

3.02 PLACING STONE

A. Locate stone as detailed on the Drawings.

B. Place stone as detailed on the Drawings and as directed by A/E or landscape architect.

C. Place soil separator fabric between stone materials and backfill as detailed on the Drawings. Allow overlap so that the fabric extends above and below granular gravel backfill.

D. Remove unacceptable and excess stones and construction debris from the site.

E. Stone step tolerances shall be heights shown on the Drawings, plus or minus 1 in. Horizontal tolerances for the step faces shall be less than 3 in. per 10 ft. laterally.

3.03 CLEANING

A. Remove and replace stone that are loose, broken, stained, or otherwise damaged. Provide new matching units and install as specified.

B. Clean stonework not less than 6 days after completion of the installation. Use clean water.

C. Upon completion of the work, remove all excess materials, debris tools, and equipment from the site. Contractor shall repair any damage resulting from stone placement operations at its expense.

END OF SECTION

SECTION 31 41 22

STEEL SHEET PILING

PART 1 GENERAL

1.01 SUMMARY

A. Provide steel sheet piling as shown and as specified. Comply with applicable provisions of Divisions 00 and 01.

1.02 SUBMITTALS

A. Shop Drawings: Submit shop drawings showing layout and materials; include pile designation, material and section properties, embedment, pile formula, hammer model, energy rating, frequency, and amplitude.

B. Make submittals in accordance with Section 01 01 00.

1.03 PROTECTION OF ADJOINING PROPERTY

A. Protect structures, underground utilities, and other construction from damage caused by pile driving operations.

B. Contractor shall notify all property owners immediately adjacent to or abutting project. An inspection of each property shall be made jointly by property owner, Contractor, and A/E to document existing conditions prior to construction as well as after construction is complete.

C. Claims of damage arising from driving or withdrawal of piling shall be the responsibility of and settled by Contractor.

PART 2 PRODUCTS

2.01 SHEET PILES

A. Steel sheet piles shall be new piling, conforming to ASTM A572, Grade 50. Sheet piles shall be standard interlocking type having positive interlocks effective in both longitudinal and transverse directions, and continuous throughout length of pile. Fabrication of piles from shorter lengths will not be permitted.

PART 3 EXECUTION

3.01 DRIVING EQUIPMENT

A. Hammer: Provide low frequency, vibratory driving hammer.

3.02 SITE PREPARATION

A. Complete excavation within area to be occupied by bearing piles before piles are driven.

3.03 PROTECTION OF PILE HEADS

A. Protect heads of piles by suitable caps, heads, blocks, mandrels, and other devices. Cut heads of steel piles square and fit with a steel driving cap. Provide driving heads, mandrels, and other devices as needed for special types of piles conforming to recommendations of pile manufacturer.

3.04 DRIVING SHEET PILING

A. Drive piling in a manner to insure perfect interlocking throughout entire length of each pile. Hold piles in proper alignment during driving by means of assembling frames or other suitable temporary guide structures. Remove temporary guide structures when they have served their purpose.

B. Any time that forward edge of sheet pile wall is found to be out of correct alignment, piling already assembled and partly driven shall be driven to required depth, and taper piles shall then be driven to bring forward edge into correct alignment before additional regular piling is assembled and driven. Maximum permissible taper in a single pile shall be 1/4 in. per ft of length.

3.05 CUTOFF

A. Cut piles at specified elevations. Length of pile cut off shall be sufficient to permit removal of damaged material.

END OF SECTION

SECTION 31 81 90

CONTAINMENT SITE OPERATIONS

PART 1 GENERAL

1.01 SUMMARY

A. Provide for an organized, nuisance-free operation of the "Navigational Dredge Spoils Containment Site" (Containment Site) that will satisfactorily protect the environment and provide for efficient operation. Comply with applicable provisions of Divisions 00 and 01.

B. Operations shall include managing spoils hauling with harbor dredging operations so that both operations occur efficiently. Operations shall include hauling of shredded wood for use during operations to improve access to spoils dumping locations within Containment Site area.

C. Site operations shall manage the spoils and shredded wood delivery into the Containment Site area so that spoils are placed to limit additional handling. Provide for a tracking pad, prevent the transport of site soils and spoils to areas outside the Containment Site and beyond the limits of construction; clean up daily the sediments and soils that have migrated beyond the construction limits and return them to within the Containment Site area.

D. Follow the storm water permit requirements. Site operations shall manage storm water, consolidation water from spoils, and erosion controls to prevent sediment transport beyond the limits of construction. Erosion occurring in areas outside the containment site area shall be repaired following significant damaging storm events and weekly, as necessary, including the installation of additional erosion control measures in accordance with Section 01 57 19.

1.02 OPERATIONS

A. Contractor shall operate the Containment Site in conformance with the contract requirements, the WDNR-Approved Exemption Request, and state and federal regulations. Operational plans, drawings, and approval letters are available from the A/E for use by Contractor.

B. At a minimum, the operations work shall include:

1. Preparation and maintenance of the project site and Containment Site area.
2. Shredded wood hauling and utilization within the Containment Site area.
3. Spoils inspection and acceptance.
4. Spoils placement, grading, and management.
5. Stormwater management and sediment/erosion control.
6. Maintenance of access roads and Containment Site disposal access.
7. Hauler vehicle assistance.
8. Litter, debris, and sediment control and cleanup.

1.03 HOURS AND DAYS OF SPOILS RECEIPT

A. The site shall be open to receive spoils whenever hauling is taking place from the Menekaunee Harbor dredging work. Hauling of spoils to the containment site is the controlling activity and work shall be coordinated with foreman controlling sediment operations at harbor.

B. Contractor shall be on-site a minimum of 30 minutes prior to receipt of spoils and shall have proper equipment operating by the time spoils are received.

C. During periods of inclement weather or other warranting conditions, Contractor shall be on-site sufficiently ahead of spoils acceptance to prepare access roads and Containment Site accessibility, and as directed by A/E.

D. Weather conditions will not be cause for extra compensation.

1.04 SUBMITTALS

A. Submit items to A/E shall be in written format, with submittal title and Contractor name prominently identified. Submittals shall be received by A/E prior to the date listed below. Submittal information shall be resubmitted to A/E within two business days of submittal information changes.

B. Personnel List: Prior to beginning operations and prior to personnel changes being made, Contractor shall submit a list of personnel scheduled to work at the Containment Site to A/E.

C. Spoils Placement Plan: Ten days prior to beginning operations at Containment Site, submit review plan for waste placement, sequencing, and stormwater and consolidation water management to A/E and Owner for review.

D. Contractor Health and Safety Plan: Five business days prior to beginning operations, submit a health and safety plan for Contractor's personnel. Owner and A/E are not responsible for Contractor personnel safety.

E. Communications Plan: Five business days prior to beginning operations, submit a communications plan. Communications plan shall include, at a minimum, contact information for the site foreman, including phone numbers, and address for all written communications. Foreman for harbor activities associated with spoils loading and hauling shall be included.

F. Emergency Plan: Five business days prior to the beginning of operations, submit a plan for communications, organization, and responsibilities for Contractor's employees to be implemented during emergencies. Emergencies include, but are not limited to: fires, medical emergencies, weather emergencies, and extended hours of operations. Include contact information in this submittal.

G. Make submittals is accordance with Section 01 01 00.

1.05 NOTIFICATIONS

A. Notifications to A/E for changes in personnel or equipment must be submitted in writing.

B. Notify A/E three business days before any planned change in designated foreman.

C. Notify A/E a minimum of 48 hours prior to any planned equipment issues, including those for scheduled repairs, maintenance, etc. Contractor shall be responsible for arranging for approved replacement equipment required to comply with contract requirements.

D. Notify A/E within eight hours of equipment being designated as out of service.

E. Notify Owner and A/E within 24 hours of proposed hauling of shredded wood from Owner's facility to Lot 24 so that Owner can provide loading operations for Contractor dump trucks.

PART 2 PRODUCTS

2.01 GENERAL

A. Provide all equipment, fuel, lubricants, and labor as required for operation of the Containment Site, including support equipment such as vehicles, fuel storage tanks, fuel pumps, hoses, electrical wiring, utility charges, and other items incidental to the operation of required equipment and the containment site.

2.02 EQUIPMENT

A. Provide equipment for use in spoils handling, soil and road grading, earth moving, stormwater management, and other duties required for operation and maintenance of the Containment Site, including, but not limited to:

1. Bulldozer for placing and shaping received dredge spoils.
2. Self-contained water (trash) pump capable of 200 gal per minute at 20 ft head, operable for 24 hours without refueling, and having sufficient suction and discharge hose to accommodate various pumping needs. A minimum of 40 ft of suction hose and 150 ft of discharge hose shall be readily available within 24 hours after measurable rain, or on request by A/E or Owner.

2.03 COMMUNICATION EQUIPMENT

A. Provide cellular telephones to allow containment site personnel to contact A/E or other designated Owner representative at all times, while on or off the site.

PART 3 EXECUTION

3.01 GENERAL

A. Execute the work in a timely and diligent manner so as to enable an uninterrupted and organized operation in coordination with the Menekaunee Harbor dredging operations.

3.02 SHREDDED WOOD HAULING AND PLACEMENT

A. Haul and utilize loads of shredded wood from Owner into Containment Site area after preparation of Containment Site area base. Spread shredded wood to uniform thickness of 3 to 4 in. across Containment Site area base prior to receipt and placement of navigational dredge spoils.

B. Haul and manage shredded wood as roadbed material to improve truck mobility within Containment Site area to access dumping locations.

C. All shredded wood located at Owner's facility (2411 Cleveland Avenue, Marinette, WI) shall be utilized during operations of Containment Site to the extent possible. Remaining shredded wood at end of spoils placement in Containment Site may be placed on top of finish spoils pile prior to placement of topsoil during restoration. Confirm with A/E prior to final topsoil placement over Containment Site.

3.03 GENERAL SPOILS PLACEMENT

A. Place spoils with the goal of achieving lowest possible final grades within the Containment Site area and to allow for final topsoil cap placement in a timely manner following completion of spoils placement. The goal is to utilize the majority of the Containment Site area footprint to limit the overall final height of the containment pile. The pile may need to be consolidated initially to facilitate access and dumping of spoils; however, following termination of spoils delivery, the resulting pile shall be graded to cover majority of Containment Site footprint in a uniform manner as indicated by the Drawings and Specifications.

B. Spoils shall be placed starting in the northern parts of the Containment Site area (filling from areas of higher elevation toward areas of lower elevation) leaving sufficient room for consolidation water and stormwater to drain from the spoils pile primarily toward the perimeter drainage swales along the south side of the Containment Site, and ultimately to the drainageway and into the existing detention basin.

C. Place spoils in a single containment pile that will grow in size and expand across the containment area as more spoils are brought to the site. Sufficient surface area shall be

maintained to direct consolidation water and stormwater toward the drainageway, while maintaining suitable access for vehicles bringing spoils to the site.

D. The spoils shall be placed as not to exceed a 4 horizontal to 1 vertical (4H:1V) slopes.

E. Daily covering of spoils is not required or necessary.

F. Construct and maintain vehicle access within the containment area to manage vehicles delivering spoils. Containment Site users shall not have to traverse exposed spoils to discharge spoils loads.

1. Shredded wood may be placed in vehicle access areas to facilitate access by spoils hauling vehicles (i.e., to prevent vehicles from becoming stuck).
2. Contractor may use aggregate to facilitate spoils hauling vehicles access to the dumping location within the Containment Site area; however minimize aggregate use and attempt to reuse aggregate as dumping locations change. Coordinate aggregate use with A/E by providing information regarding selected materials and its proposed use. A/E and Owner understand that soft access conditions may occur following precipitation events and aggregate use may be necessary.

3.04 SITE MAINTENANCE

A. Maintain site in a neat and orderly manner at all times. Provide maintenance including, but not limited to, general road grading, site drainage, stabilization and restoration of site erosion problems, and any other such needs within the capabilities of the specified equipment and labor.

B. Divert stormwater away from areas containing spoils and whenever practicable within the containment area. Refer to applicable WDNR technical specifications. Water shall be directed toward constructed drainage courses. Maintain site and erosion control BMPs to control sediment transport and erosion. Water coming in contact with spoils shall be considered “leachate” and this water shall not be discharged outside of the designated limits of construction, to the delineated wetland, or to offsite areas.

C. Do not let water pond for long periods of time. Contractor shall have pump(s) available and shall pump ponded water from depressions and correct depressed areas. A minimum slope shall be maintained across the Containment Site to direct water to the perimeter drainage swales and to the detention basin.

D. Maintain site in an erosion-free condition in all site drainage features, including, but not limited to, slopes, drainage swales, drainageway, and detention pond. Install Contractor-furnished erosion control materials (see Sections 01 57 19 and 31 05 10). Construct and maintain existing site access road and new access road into containment site, and vehicle access to spoils dumping locations with shredded wood furnished by Owner or aggregate materials provided by Contractor.

3.05 ROAD MAINTENANCE

A. Construct and maintain road access into Containment Site to facilitate spoils dumping. Road shall be constructed of materials that will not excessively rut or settle, or that are excessively slippery, and in accordance with Drawings and Specifications.

B. Maintain existing site entrance and access road (starting at end of Murray Street cul-de-sac and up to connection with, and including, access road constructed to access containment site area. NOTE: Site access road is used by other adjacent property owners and their contractors (shared access road along south side of project property).

3.06 EQUIPMENT MAINTENANCE AND FUELING

A. Contractor may perform equipment maintenance and fueling on-site, but in such a fashion as not to delay incoming spoils shipments or placement. Follow best management practices for spill containment. Contractor shall be responsible for any remedial investigation or remedial action required due to contamination caused by spillage or leakage of Contractor's equipment during operations, maintenance, repair, or fueling.

3.07 LITTER PREVENTION AND CONTROL

A. Take prompt measures to help prevent, control, contain, and collect Contractor-generated waste and debris to satisfaction of A/E and Owner.

3.08 FIRE CONTROL

A. Take suitable precautions to prevent fires and control them if they start. Fire extinguishers shall be furnished and maintained on all equipment and in any temporary personnel or storage buildings. If a fire breaks out in an area under Contractor's control, Contractor shall immediately contact local fire protection agency and then notify A/E and Owner. No additional compensation will be allowed for fire control operations. Contractor shall compensate fire department(s) for services required to extinguish fires, as applicable.

3.09 DUST CONTROL

A. Provide dust control measures as appropriate following WDNR guidelines presented in WDNR technical specifications. Dust control shall include at a minimum, application of water on the access roads and within the containment site. Chemicals or oils shall not be used as a dust control agent. Shredded wood / wood chips may provide relief from dust. No additional compensation will be allowed for dust control operations.

3.10 FOUL WEATHER OPERATIONS

A. Provide for site and Containment Site access in all weather conditions to the extent practicable. Provide for proper drainage on all roads, excavations, soil stockpiles, and containment site area spoils dumping locations to minimize traffic problems and insure accessible operations. No additional compensation will be allowed for foul weather operations.

3.11 COLD WEATHER OPERATIONS

A. Provide for proper protection of equipment to enable continued operations during cold conditions. No additional compensation will be allowed for cold weather operations.

3.12 GRASS AND WEED CONTROL

A. Contractor is not responsible for mowing or cutting vegetation during site operations following completion of proper clearing and grubbing, and site preparation activities in accordance with Sections 01 57 19 and 31 05 10.

END OF SECTION

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SECTION 31 81 92

CONTAINMENT SITE CLOSURE AND EXIT

PART 1 GENERAL

1.01 SUMMARY

A. Provide containment site closure and exit. Comply with applicable provisions of Divisions 00 and 01.

1.02 NOTIFICATION

A. Notify A/E a minimum of 7 days prior to the anticipated completion of spoils containment and site operations, and to schedule project close-out meeting at the site on last day of site operations.

1.03 SUBMITTALS

A. Punch-List Completion Schedule: Submit a schedule for correction of agreed upon “punch-list” items in writing within two working days of project site close-out meeting between Contractor, A/E, and Owner.

B. Make submittals in accordance with Section 01 01 00.

PART 2 (NOT USED)

PART 3 EXECUTION

3.01 GENERAL

A. Complete the agreed upon punch-list in a timely and diligent manner.

3.02 SITE CONDITIONS

A. A joint inspection of the containment site in its entirety shall be made by Contractor, A/E, and Owner at a date prior to end (but within 2 days of anticipated end) of Contractor's project site operations.

B. A “punch-list” of items to be completed by Contractor will be identified and submitted to Contractor in writing by A/E. The list will identify tasks and expected completion dates prior to the overall exit date. Owner reserves the right to add items to “punch-list” throughout the exit process.

C. Prior to the final exit date, a final inspection between A/E and Contractor shall take place to ensure all “punch-list” items have been completed to the Owner’s satisfaction.

D. Contractor equipment shall be removed on final exit date or shortly thereafter, as agreed to with Owner.

3.03 RESTORATION OF DISTURBED AREAS

A. All areas disturbed by Contractor shall be re-graded and restored to Owner's satisfaction in accordance with Sections 31 22 00 and 32 92 00 prior to Contractor’s departure. This includes, but is not limited to, topsoil stockpile location, truck cleaning areas (tracking pad), and access roads. Refer to WDNR technical specifications.

3.04 ROUTINE OPERATIONS

A. Once an exit date is established, Contractor shall continue required operations contract on a regular basis until the exit date. These activities shall not be delayed unreasonably.

END OF SECTION

SECTION 35 20 13

DREDGING

PART 1 GENERAL

1.01 SUMMARY

A. Provide mechanical or hydraulic dredging as shown and as specified. Comply with applicable provisions of Divisions 00 and 01.

1.02 CLASSIFICATION

A. Materials encountered under this work will be classified as the following for the purposes of this project:

1. "Environmental Dredge Spoils" are dredge spoils that are contaminated at levels greater than the Midpoint Effect Concentration (MEC) as outlines in the *Consensus-based Sediment Quality Guidelines*, prepared by the WDNR Contaminated Sediment Standing Team, December 2003 (WT-732 2003). A copy of this document can be found at http://dnr.wi.gov/topic/brownfields/documents/cbsqg_interim_final.pdf. These materials are to be removed and disposed at the designated landfill.
2. "Navigational Dredge Spoils" are the remaining dredge spoils to be removed from the harbor to achieve the final desired harbor depths. Navigational dredge spoils with arsenic levels below 6 ppm can be sent to the Lot 24 Navigational Dredge Spoils Containment Site. Navigational Dredge Spoils with arsenic levels greater than 6 ppm but less than the MEC are to be disposed at an approved landfill if not reused within the harbor.
3. "Beneficial Reuse Dredge Spoils" are the portion of the Navigational dredge spoils which will not leave the harbor site. These materials will be maternal categorized as "Navigational Dredge Spoils" with regards to contamination levels (less than MEC) and relocated to the east end of the harbor to achieve desired final grading.

B. Dredge spoil sediment contaminate sample summary table is included in Appendix A.

C. Dredge spoil physical characteristic summary table is included in Appendix B.

D. See "Quality Assurance Project Plan (QAPP) and Dredge Handling Plan" in Appendix C for detailed testing and handling requirements. "QAPP and Dredge Handling Plan" included in Appendix C is a draft. Contractor shall provide any additional information required to finalize this document for approval by all parties outlined in the document. No work shall commence prior to finalization and approval this document.

1.03 SUBMITTALS

A. Dewatering Plan: Within 10 days after Notice to Proceed, submit the proposed dewatering plan for submittal of Chapter 30 dredging permit application to WDNR. Contractor is responsible for developing a plan to meet the discharge requirements outlined in WWTP Agreement Letter (Appendix E) or WDNR Carriage and Interstitial Water Requirements (Appendix F) depending on selected method for wastewater disposal. Include, at a minimum, the following:

1. Dewatering system design/plans and specifications.
2. Wastewater discharge location (include both groundwater and surface water locations if utilized).
3. Pump information.
4. Piping layout.
5. Instrumentation to be used.
6. Equipment to be used.

7. Any chemical that might be used.
8. Anticipated flow rates.
9. System monitoring plan.
10. Water treatment plan (if WWTP is not used).
11. Sampling and testing plans.

B. Work Plan: Prior to beginning work, submit for information only a Work Plan for approval that includes, at a minimum:

1. Complete schedule of dredging operations including start and end dates for each dredging area.
2. Dredging methods to be used and descriptions of equipment to be utilized for work to be completed. Include process for collecting and treating carriage and/or interstitial water. Include methods for achieving desired dredge depths and tolerance quality control. Include methods for backfilling east area of harbor with excess dredge spoils.
3. Sequencing plan including order or areas to be dredged, coordination with shoreline treatment, and methods for dredging around shallow waters, sea walls (or other shoreline stability structures), and critical structures in the harbor.
4. Erosion control and security details around staging and dewatering areas.
5. Waterway markers, boat traffic maintenance plan, and protection of commercial/recreational watercraft during dredging.

C. Weekly Reports: Submit weekly reports detailing hours of excavation and quantities of sediment removed.

D. Carriage & Interstitial Water from Dredging Operations General Permit Discharge Monitoring Reports (DMRs). These are only required if wastewater from dredge operations is not sent to the WWTP for treatment.

E. Make submittals in accordance with Section 01 01 00.

1.04 MEASUREMENT AND PAYMENT SURVEYS

A. Contractor shall conduct pre and post dredge bathymetric surveys to document net quantities removed or placed by Contractor and confirm target elevations for both navigational and environmental dredging section of the harbor. Surveys shall be conducted:

1. Any time dredge spoil classification transitions from environmental to navigational.
2. After final dredge depth has been achieved (prior to backfilling with beneficial reuse material).
3. After placement of beneficial reuse material.
4. After Additional Environmental Dredge/Disposal (if additional dredging is required).

B. Contractor shall arrange and pay for services of a Wisconsin-licensed Registered Land Surveyor (RLS) or a Professional Engineer who is independent of Contractor and Owner to complete measurement and payment surveys.

C. Bathymetric surveys shall be completed using standard hydrographic survey methods.

D. Bathymetric survey data will be used to compute final pay volumes for NAVIGATIONAL DREDGING/DISPOSAL and BENEFICIAL REUSE DREDGING.

E. Contractor shall provide weigh tickets from the designated landfill for tracking and payment.

1.05 PERMITS

A. Owner will apply for dredging, interstitial water, construction site stormwater discharge, waterway marker, and shoreland erosion control permits and provide copies to Contractor. Contractor shall post permits on site where directed by Owner. Permits shall be posted at

least 5 days prior to beginning work and shall remain in place at least 5 days after completion of work.

B. Contractor shall comply with all conditions of permits including, but not limited to, monitoring, limitations, and reporting requirements related to total suspend solids and all other parameters and equipment decontamination requirements.

C. Obtain and pay for all other necessary licenses and permits, and comply with applicable Federal, State and local laws and regulations. Display permits where directed by Owner.

1.06 ACCESS

A. Owner will furnish access easements to dredge site and any other easements required for construction.

1.07 PROTECTION

A. Provide sufficient barricades and protective devices around excavations to safeguard against injury. Provide and maintain sufficient safety lanterns at night. Comply with Section 31 05 10.

B. Comply with applicable Federal, State, and local laws and regulations concerning environmental pollution control and abatement.

1.08 EXPLOSIVES

A. Use of explosives is not permitted.

PART 2 (NOT USED)

PART 3 EXECUTION

3.01 DREDGING EQUIPMENT

A. Types and capacities of equipment suitable for use at the site and production rates to be obtained are the responsibility of Contractor.

3.02 LAYOUT OF WORK

A. Lay out work from Owner-furnished baseline(s) and benchmark(s) as indicated on the Drawings and be responsible for all measurements. Furnish stakes, templates, platforms, equipment, range markers, and labor as required to lay out work. Contractor shall be responsible for execution of work to such lines and grades and shall monitor grades daily.

B. Maintain and preserve stakes and other marks until authorized to remove them. Marks destroyed by Contractor or through its negligence prior to their authorized removal may be replaced by Owner at its discretion; expense of replacement will be deducted from amounts due or to become due Contractor.

3.03 DREDGING

A. Provide dredging to depths and widths as shown. Blend slopes of dredged area into existing bank slopes.

B. Provide and maintain necessary temporary access roads, haul roads, signal lights, bouys, and environment protection; costs shall be incidental to contract.

C. Dredge depth tolerance shall be elevation shown on Drawings, plus or minus 3 in. Horizontal dredging tolerance shall be plus or minus 2 ft.

D. In general, spoils with more than 30% fines (particles smaller than the #200 sieve) shall be anticipated to be Environmental Spoils and disposed of accordingly. Remaining spoils with less than 30% fines shall be anticipated to be Navigational Spoils and disposed of/reused accordingly. Samples may be collected by A/E and tested for grain size and contaminants at any time during construction for compliance.

E. Owner will be responsible for collection of confirmation sediment samples during dredging operations. Confirmation sampling will be completed as outlined in the "Quality Assurance Project Plan (QAPP) and Dredge Handling Plan" in Appendix C. Contractor shall allow access to harbor for this sampling. When Contractor, working with Owner's site representative, believes that the contaminated sediment has been removed from an area, Owner's representative will begin confirmation sampling. It is intended that as 100 foot by 100 foot areas are remediated, samples will be collected. Sample results will be available within 10 calendar days of collection. Contractor may be required to return to a previously dredged area to over-excavate and remove additional contaminated material pending sample results. In areas where Beneficial Reuse Dredge Spoils are being placed, perform confirmation sampling 1-ft prior to final fill grades to ensure places material is within acceptable limits for contamination outlined under the “Classification” article in Part 1 General, above.

3.04 SEDIMENT DEWATERING

A. Contractor is responsible for completing any required treatability testing on spoils required to determine dewatering methodology and plan.

B. Background and elutriate testing was performed on water and sediments from the harbor to determine the base contamination and anticipated wastewater contamination levels during operations, respectively. The results of these tests are included in Appendix D.

C. All carriage and/or interstitial water must be collected and treated from dredge spoils to be disposed off-site prior to discharge of wastewater into Harbor. Use of sealed or watertight barges and buckets may be required to prevent discharge of untreated wastewater from the contaminated sediment if mechanical dredging is chosen as the preferred alternative. Contractor is responsible for construction of dewatering pad(s) and barge offloading structure with containment to protect surface and groundwater discharge for any mechanical dredging and dewatering operations as necessary.

D. Beneficial Reuse Dredge Spoils can be transported directly to final placement location in Harbor without dewatering. Beneficial Reuse Dredge Spoils must be handled with care as to not create additional adverse water quality conditions. Provide additional turbidity barriers around both the dredging and placement locations as necessary. If visual turbidity problems occur, placement shall cease until other mothods are evaluated to continue with placement. Use of sealed or watertight barges may be required to prevent discharge of suspended solids from the sediment during transport if mechanical dredging is chosen as the preferred alternative.

E. Sediment from dredge operations shall meet the following criteria before considered “dewatered” and is allowed to be transferred off-site for disposal.

1. Sediment must contain no free liquids (passes *Paint Filter Liquids Test*, U.S. EPA SW846 Method 9095B).
2. Sediment must be able to support its own weight.
3. Sediment must be able to support the weight of the material placed over it.
4. Sediment must be capable of being worked by the Disposal Site’s low ground pressure bulldozers.

F. The City of Marinette Wastewater Treatment Plant (WWTP) has agreed to accept any wastewater from dredging. The agreement outlining conditions and limits for the WWTP is included in Appendix E. Wastewater shall be sampled by Contractor as outlined in the agreement letter.

G. Contractor shall determine dewatering method that meets the WDNR WPDES (WI-0046558-5) Carriage and Interstitial Water from Dredging Operations General Permit (if WWTP option is not used). Permit will be applied for after Notice to Proceed. Limits and requirements required by the WDNR are included in Appendix F.

H. Comply with the erosion control provisions of Section 01 57 19.

3.05 DISPOSAL

A. Dispose of dredged material in designated disposal areas based on material classification outlined. Distribute dredged material at disposal area as shown on Drawings.

B. Contractor may propose a different licensed landfill disposal site for Environmental Dredge Spoils. Contractor shall be responsible for providing all necessary sampling and testing required for landfill profile approval process. Owner must approve any proposed disposal site other than approved landfill in Menominee, Michigan.

C. Remove and dispose of snags and fallen trees that will extend above top of spoil.

D. In the event disposal areas have insufficient capacity due to excessive overdredging, Owner will be responsible for obtaining additional disposal areas.

E. Leakage or spillage of hauled materials on to public roads and streets will not be permitted, and if unavoidably done, shall be removed daily in a manner acceptable to Owner. Material boxes of hauling equipment shall be of watertight construction and shall not be loaded over their rated capacity; nor shall loads exceed limits of local thoroughfares over which they operate. Comply with laws and regulations pertaining to operation of vehicles.

3.06 POST-CONSTRUCTION CLEANUP

A. Obliterate all signs of temporary construction facilities such as haul roads, work areas, buildings, stockpiles of excess or waste materials, or other vestiges of construction. Disturbed areas shall be graded and filled as required.

3.07 FINAL EXAMINATION AND ACCEPTANCE

A. Post dredge sampling plan is included in the “Quality Assurance Project Plan (QAPP) and Dredge Handling Plan” in Appendix C. Post-dredge samples will be collected at locations where a contaminant was present at levels greater than the MEC during pre-dredge sampling. If contaminants are found to be present in levels exceeding the TEC the results will be evaluated to determine an acceptable solution, which must be approved by the WDNR prior to implementation.

B. As soon as practicable after completion or completion of any section that will not be subject to further operations, the work shall be examined by survey, sounding, or by sweeping. If shoals, lumps, or other lack of depth are disclosed by this examination, Contractor will be required to remove same by dragging bottom or by dredging; however if bottom is soft and shoal areas are small and form no material obstruction, removal of such shoal may be waived.

C. A/E shall be notified when surveys, soundings, and/or sweepings will be made, and shall be permitted to accompany survey party. When an area is found to be in a satisfactory condition, it will receive final acceptance. Should more than one survey, sounding, or sweeping operation over an area be necessary by reason of work for removal of shoals

disclosed in prior examination, the cost of second and subsequent survey, sounding, or sweeping operation shall be borne by Contractor.

END OF SECTION

APPENDIX D

Wastewater Treatment System Monitoring Plan

WASTEWATER TREATMENT SYSTEM MONITORING PLAN

Wastewater Treatment System Monitoring Plan

Site water that has been treated utilizing Sevenson's Waste Water Treatment System (WWTS) will be discharged into the City of Marinette's POTW.

After processing an initial volume of 18,000 gallons, the volume will be retained in an effluent storage tank while samples are taken and analyzed. Influent and effluent samples will be analyzed as specified in the City of Marinette's Water Utility's Letter of Agreement, dated March 25, 2014 and noted below:

Arsenic	Cadmium	Chromium	Copper	Lead
Mercury	Nickel	Selenium	Silver	Zinc
Cyanide	Oil & Grease	Phenols	pH	BOD
TSS	PHOS			

Schedule of Sampling and Analysis

Sevenson's Wastewater Treatment sampling and monitoring program is required for compliance with the aforementioned Letter of Agreement and for day-to-day control of the treatment plant.

Sevenson will collect samples and conduct the analyses for the following reasons:

1) To form a rational basis for controlling the treatment processes.
2) To define the operational efficiency of the plant.
3) To satisfy the discharge reporting requirements of the Water Utility's Letter of Agreement. (Attached)

Sampling

Grab samples will be utilized to sample influent and effluent flows. All samples will be taken by Sevenson staff. The influent samples will be taken at the entrance to the first Influent Equalization Tank. Effluent samples will be taken at the Bag Filter Effluent Sample Port.

Sevenson will conduct all analyses for the treatment plant. All samples will be properly preserved and sent to a City of Marinette Wastewater Utility approved laboratory.

All samples will be analyzed in accordance with the current edition of Standard Methods or other appropriate, recognized testing standard.

Analysis Schedule

Wastewater shall be sampled to confirm compliance with the discharge limits outlined in the Menekaunee Harbor Improvements WWTP Agreement Letter, prior to any discharge into the local POTW. Following the initial analysis and approval, wastewater will be sampled according to the schedule below for discharge limit compliance throughout the duration of the project:

1. Two times on two different days during the first calendar week of discharge.
2. Once per week during the second through fifth calendar weeks of discharge.
3. Once per month after the fifth week for the remaining duration of the project.
   Monthly frequency only applies if data indicates substantial compliance in all prior analyses.

All analytical results will be provided to the Utility Operations Manager for the City of Marinette and the Project Engineer. If at any time during the project, Effluent analytical fails to meet the required discharge limits, the WWTS will be shut down immediately for retesting and re-evaluation of treatment and disposal procedures. The Marinette Utility Operations Manager and the Project Engineer will be notified.

Water Meter

Sevenson is proposing to utilize a Seametrics FT415 Flow Meter to measure flow to the WWTP for payment and monitoring purposes. The specification sheets for this meter are provided as attachments for your review and approval.

ATTACHMENTS

Attachment 1	Marinette Water Utility Letter – March 25, 2014
Attachment 2	Seametrics FT400-Series Specification Sheets

Marinette Water Utility

501 Water Street * Marinette, Wisconsin 54143
Phone (715) 732-5180 * Fax (715) 732-5194

RECEIVED
MAR 28 2014
AYRES ASSOCIATES

March 25, 2014

Re: Menekaunee Harbor Improvements – WWTP Agreement Letter

Dear Bidder for Menekaunee Harbor Improvement Project:

The City of Marinette will allow for wastewater generated from dredging operations associated with the Menekaunee Harbor Improvement project to be discharged into the sanitary sewer system for treatment at the City's wastewater treatment plant (WWTP). This agreement letter is only valid for the 2014 calendar year. This letter and enclosed exhibit outline the discharge requirements for treatment by the WWTP. The cost to the contractor for treatment of wastewater from the dredging will be $6.69/1000 gallons of water discharged. Contractor shall provide City approved flowmeter for measuring wastewater volume discharges.

The local limits for the Marinette Wastewater Treatment Plant are outlined in Table 1.

Table 1: City of Marinette Wastewater Treatment Plant Local Limits

METALS		
Arsenic	mg/L	0.10
Cadmium	mg/L	0.14
Chromium	mg/L	2.41
Copper	mg/L	1.40
Lead	mg/L	2.02
Mercury	mg/L	0.01
Nickel	mg/L	1.35
Selenium	mg/L	0.12
Silver	mg/L	0.50
Zinc	mg/L	2.25
GRAB SAMPLES		
Cyanide	mg/L	0.19
Oil & Grease	mg/L	100
Phenols	mg/L	25
pH		6.0-9.0
SURCHARGEABLES		
BOD	mg/l	300
TSS	mg/l	350
PHOS	mg/l	14.5

Wastewater shall be sampled to confirm compliance with the limits outlined in Table 1 prior to any discharge into the system. Following initial testing results, wastewater will be sampled at the below schedule for compliance throughout the duration of the project:

1. Two times on two different days during the first calendar week of discharge.
2. Once per week during the second through fifth calendar weeks of discharge.
3. Once per month after the fifth week for the remaining duration of the project. Monthly frequency only applies if data indicates substantial compliance in all prior testing.

Provide all test results to Utility Operations Manager and Project Engineer. If at any time during construction a test fails to meet the required local limits, discharge into system will be immediately stopped for re-testing and re-evaluation of treatment and disposal procedures. Contact Utility Operations Manager and Project Engineer in the event of a failed water quality test.

A pump station is located at the south side of the Ogden Street Bridge. No wastewater will be permitted to be discharged north of the pump station. Wastewater may be discharge into the manholes indicated on the drawing included in Exhibit 1. Discharge will not be permitted in any locations other than the four manholes identified in Exhibit 1 without written permission from WWTP and Project Engineer. Two of the manholes shown are located on property owned by EFX, Inc. A written agreement with property owner will be required in order to discharge into any manhole located on private property.

Contractor shall provide manhole cover to allow wastewater discharge and restrict unauthorized access or disposal into sewer. Manhole cover shall also serve to minimize odor release from sewer.

Traffic on Ogden Street shall remain open throughout construction. Contractor shall provide signage and protection for any pipes or hoses across five traffic lanes.

Provide wastewater monitoring, treatment, and disposal plan to Utility Operations Manager and Project Engineer for review prior to start of construction.

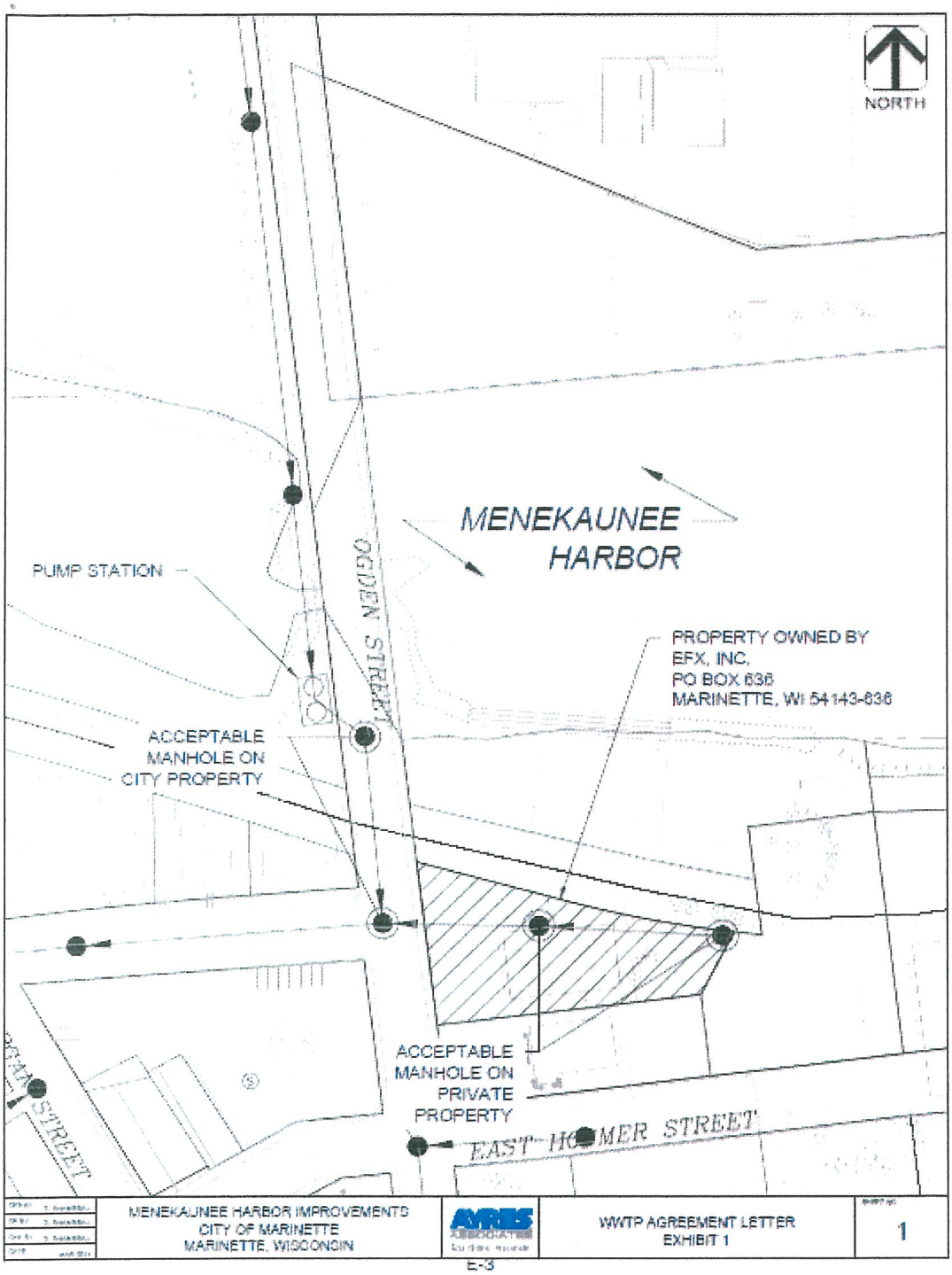
Sincerely,

City of Marinette Water & Wastewater Utility

Warren C. Howard

Warren Howard
Utility Operations Manager
715.732.5192
whoward@marinettewater.com

Enclosure: Exhibit 1



Steven C. Shaw, P.E.

Sevenson Environmental Services, Inc.
1105 North Point Blvd., Suite 327
Baltimore, MD 21224

Cell: (443) 375-1678

FT400-SERIES
Rate/Total Indicator

ISO 9001:2008 CERTIFIED COMPANY

FEATURES

- Simple Setup
- Battery (FT415) or Loop Powered (FT420)
- Remote or Flow Sensor Mounted Indicator
- Rugged Metallic Housing
- Non-volatile Memory

APPLICATIONS

- Water Treatment
- Water Utility
- Industrial Chemical Handling



FT420



FT415

GENERAL INFORMATION

The FT400-Series flow computers are microcontroller-based indicator/transmitters that display flow rate and total and provide output signals. The FT415 is battery-powered and provides a scalable pulse output. The FT420 is powered by external DC voltage and has both pulse and 4-20 mA analog outputs. When the FT420 is being used in the 4-20 mA mode, it is a “two-wire” or “loop-powered” device, meaning that the 4-20 mA output signal doubles as its power supply.

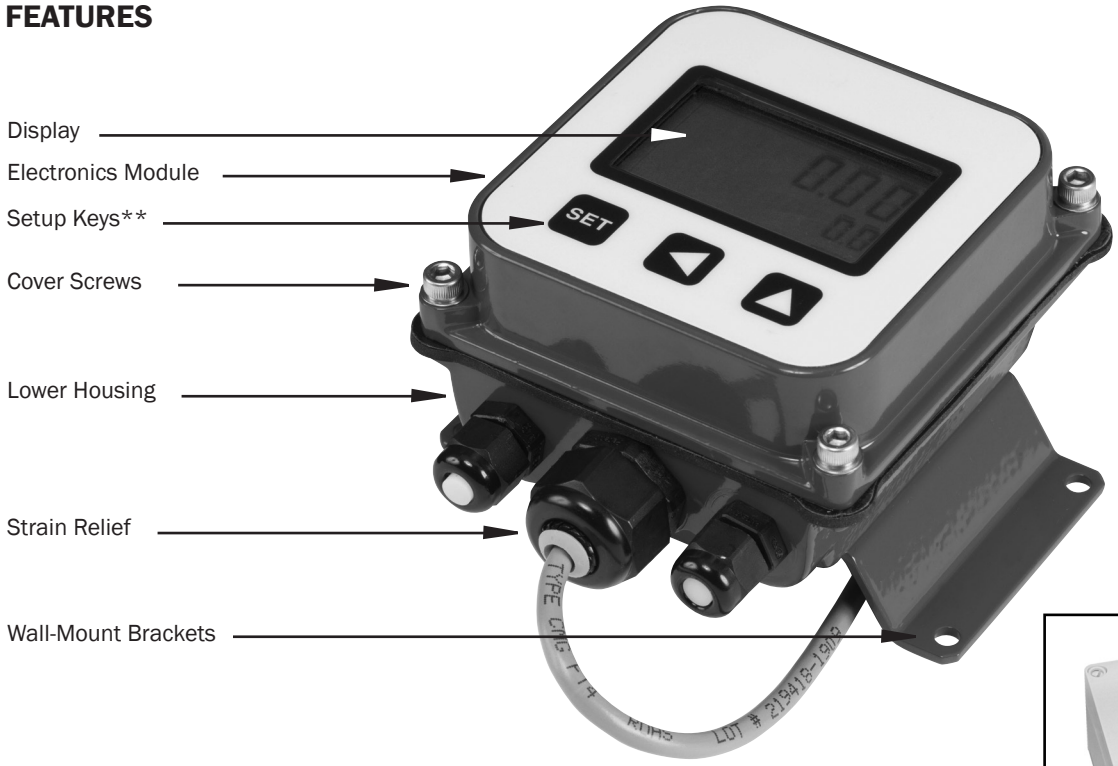
The addition of a dual-relay output board (FT420 only) allows for certain applications requiring contact output isolation (e.g., certain metering pumps and water treatment controls). Dual solid state relays provide exactly the same pulse output as the standard unit, and each can signal one external device. A non-resettable total is also available. The FT420 can be ordered in a plastic enclosure with a 115 Vac power supply for use with mechanical meters, or with a built-in 115 Vac/12-24 Vdc dual power supply for magmeters.

Both the FT415 and the FT420 can be factory-mounted on the meter (-M) or remotely wall mounted with the brackets provided (-W). The FT420 is also available as a panel mount (-P) with an open back for easy installation in the user’s own electrical enclosure. Most FT400’s can be converted from wall-to-meter or meter-to-wall mount configurations after installation if needed.

Housings for the -W and -M models are rugged cast aluminum, potted and gasketed for maximum environmental protection. A membrane keypad allows settings to be changed without removing the cover. (Password protection, a standard feature on the FT420, can be used to prevent settings from being changed.)

FT400-SERIES
Rate/Total Indicator

FEATURES



**The FT420 includes password protection for tamper prevention when needed



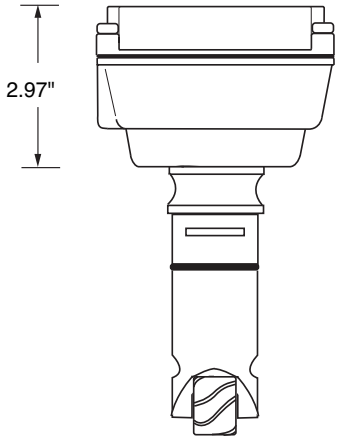
FT420 with -27 or -65 Option

SPECIFICATIONS*		FT415	FT420
Power		Lithium "C", 3.6 Vdc, replaceable. Estimated life is 3-5 years depending on usage.	12-30 Vdc, 4mA (4-20 mA when loop-powered)
Display	**Rate**	6-digit autorange, 1/2" character height	6-digit autorange, 1/2" character height
	Total	8-digit, 5/16" character height	8-digit, 5/16" character height
Outputs	**Current Sinking Pulse**	Scaled Pulse output (0.1 sec duration 6.1 Hz max) (or High Alarm output or Low Alarm output)	
		Sensor pass-through Pulse output (unscaled)	
	Analog	None	4-20 mA loop; 24-30 Vdc
Pulse Output Range		0.1 - 9999999.9 units/pulse	0.1 - 9999999.9 units/pulse
Input		Micropower GMR Sensor (square wave)	5V pulse or contact closure
Input Range		1.0 - 150 pulses/second	1.0 - 1,500 pulses/second
K-Factor Range		.001 - 99999.999	.001 - 99999.999
Flow Alarm Output Range		.01 - 999999.99	.01 - 999999.99
Operating Temperature		-30˚ to 65˚ C (-22˚ to 148˚ F)	-30˚ to 65˚ C (-22˚ to 148˚ F)
Environmental		NEMA 4X, IP66	NEMA 4X, IP66

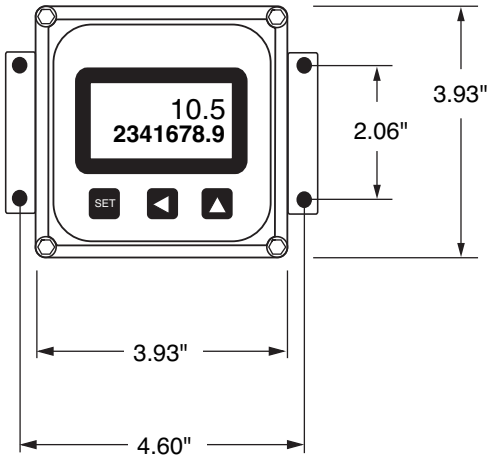
**Specifications subject to change • Please consult our website for current data (www.seametrics.com).*

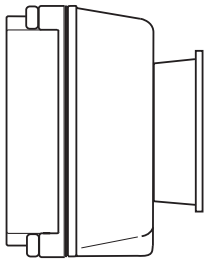
DIMENSIONS

FT415/FT420 Sensor Mount

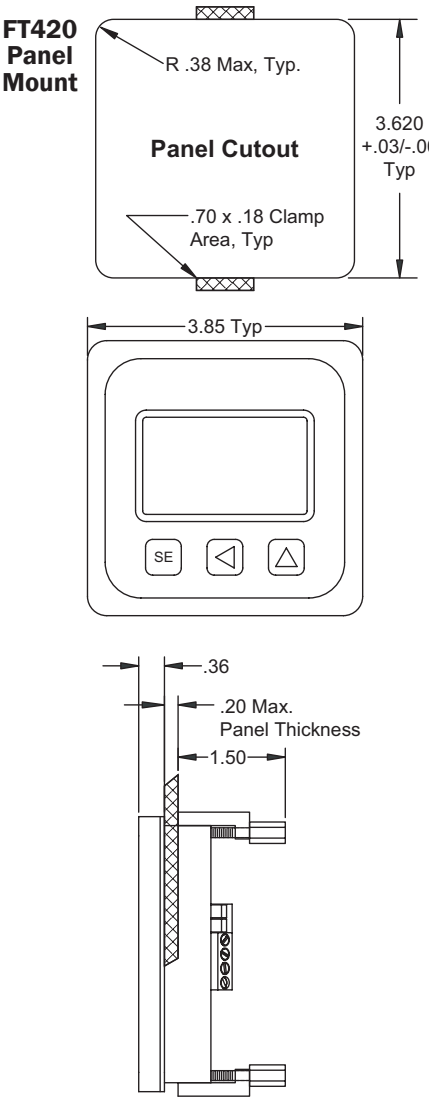


FT415/FT420 with Wall Mount Brackets

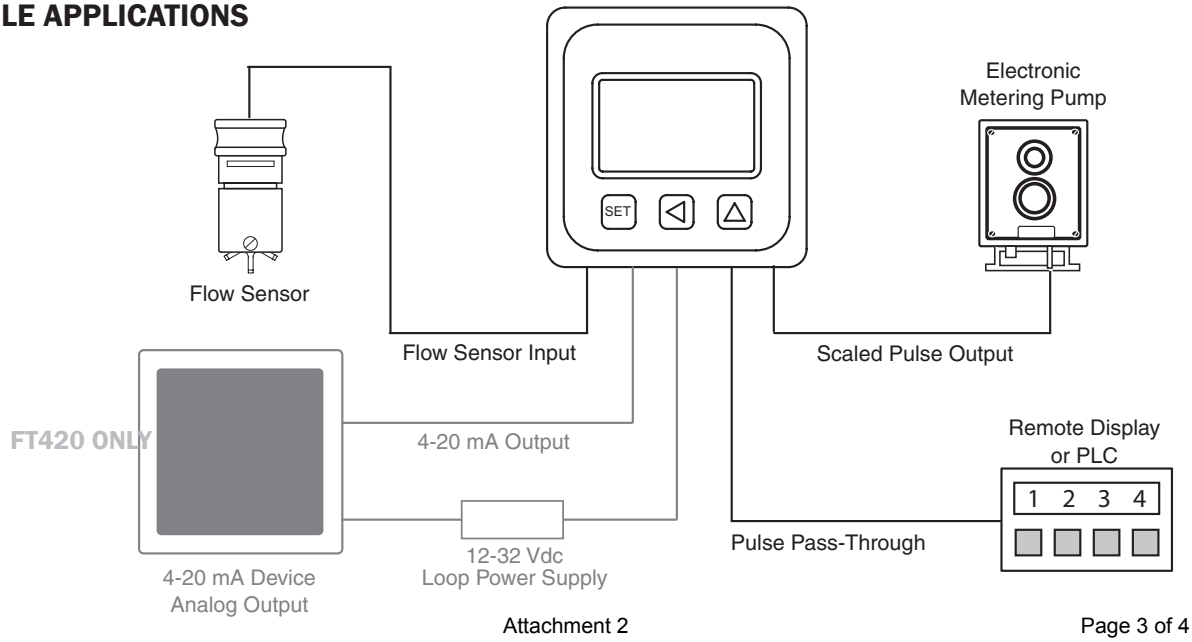




FT420 Panel Mount



POSSIBLE APPLICATIONS



FT400-SERIES
Rate/Total Indicator

HOW TO ORDER

MODEL

Battery-powered indicator = **FT415**

Loop-powered indicator/transmitter = **FT420**

MOUNTING

Premounted on meter = **M**

Wall mount = **W**

Panel mount = **P**

OPTIONS

Built-in 115 Vac/12-24 Vdc dual power supply (FT420W only, use with magmeters) = **-27**

Tamper-evident = **-32**

Non-resettable total = **-64**

Built-in 115 Vac power supply (FT420W only, use with mechanical meters) = **-65**

Dual relay output (FT420 only) = **-98**

ACCESSORIES

Data logger (wall mount) = **DL76W**

Power converter, plug-in, 115 Vac, 24 Vdc = **PC3**

Dual power supply, plug-in, 115 Vac, 12/24 Vdc = **PC42**

Protective cover, hinged = **101949**

Mounting kit, wall-to-meter conversion = **102632**

Mounting kit, meter-to-wall conversion = **102633**

LMI pump power cable = **100013**

LMI pulse out cable = **100039**

CONTACT YOUR SUPPLIER

APPENDIX E

Waste Management Agreement

July 2, 2014

Ken Paisley
Sevenson Environmental
2749 Lockport Road
Niagara Falls, NY 14305
716-609-1466
kpaisley@sevenson.com

Project: Menekaune Harbor 2014 Project - 30,000 tons

Dear Ken:

Waste Management of Wisconsin is pleased to provide you with pricing for disposal per your request. Based upon the information provided, the following summarizes our quotation.

DISPOSAL FACILITY:

Menominee RDF
W611 Elmwood Road
Menominee, MI 49858

WASTE STREAMS

Waste Description	Dredging Material
Disposal Method	Direct Landfill
Estimated Volume	30,000 tons
Disposal Price	$26.00 per ton
MI Tax	$0.36 per ton
Landfill Environmental Fee	Waived
Disposal Fuel Surcharge Fee	Waived
Profile Approval Fee	Waived
Dig out Fee (If needed)	$60.00/Load
Washout Fee (If needed)	$100.00/Load
Certificate of Burial/Destruction (If needed)	$55.00/Each Event
Transportation	N/A

ANALYTICAL TESTING REQUIREMENTS:

City of Marinette is responsible for testing; work was contracted through Ayers & Associates
Submit a completed Waste Management Generators Non-Hazardous Waste Profile with current Analytical Testing Profile available online at www.wmsolutions.com

ESTIMATED COSTS:

Disposal Rate	$26.00
MI Generator Tax - Per Ton	$0.36
Total with Tax	$26.36
Estimated tons	30,000
Total Disposal Landfill Only	**$790,800.00**

SPECIAL CONDITIONS:
Waste must meet acceptability criteria at the site and comply with local, state and federal regulations, as well as the sites permit requirements. Pricing is contingent upon site and/or sample evaluation and approval.

Strength and moisture requirements are:

1. No free liquid
2. Able to
    a. Support its own weight;
    b. Support the weight of material placed over it;
    c. Be capable of being worked and managed by the Disposal Site's low ground pressure bulldozers.
    d. Minimum unconfined compressive strength of 0.8 tons per square foot;
    e. A minimum cohesive strength of 800 pounds per square foot;
    f. A minimum short term frictional strength of 25 degrees;
    g. Defined combinations of cohesive and short term frictional strength that provide a factor of safety for the landfill slopes as determined through slope stability modeling at least equivalent to (b) or (c).

Characterization testing of the dredged sediments shall include percent solids/moisture content (ASTM D2216 or D2974); grain size distribution (ASTM D422); liquid limit, plastic limit, and plasticity index of soils (ASTM D4318); and hydraulic conductivity testing (ASTM D5856 or D2434). Testing shall be conducted at a minimum frequency of one sample every 5,000 cubic yards. Pricing is contingent upon site and/or sample evaluation and approval.

Pricing is open for consideration for a period of 30 days. Upon acceptance, pricing remains in effect up to and including 60 days from the date of the quote. Pricing based solely on the information available at this time. Additional information may be required prior to approval.

All pricing is contingent upon profile approval (physical, chemical and regulatory evaluation of material).

Payment terms are net 30-days from receipt of invoice. Late fees apply on payments received after thirty days at an accrual interest rate of 1.5% per month. Customers that do not have approved credit with Waste Management must submit an up to date credit application.

Waste Management of Wisconsin wishes to thank you for allowing us to quote on your disposal needs.

Please do not hesitate to contact me at the phone number below with any questions you may have or if you require any further assistance.

Sincerely,

Jim Barnes
Industrial Account Manager
(920) 539-1167
jebarnes@wm.com

For further assistance:

WM – Industrial Landfill Sales
Technical Service Center
W132 N10487 Grant Dr
Germantown, WI 53022
1-800-963-4776 Toll Free
1-866-800-2591 Fax

Paisley, Ken

From: Barnes, James <JEBarnes@wm.com>
Sent: Wednesday, July 02, 2014 6:56 PM
To: Paisley, Ken
Subject: RE: Menekaunee Harbor - City of Marinette, WI

Ken,

Unless WDNR specifies additional testing requirements and frequency, all WM requirements have been fulfilled. For wood WM will need a weathered wood profile completed, no testing required. If it can fit in a truck there is no size requirements.

wood debris from pilings

Please let me know if you have additional questions.

Thank you,

Jim Barnes
Industrial Account Manager
Manufacturing & Industrial
jebarnes@wm.com

Waste Management
W132 N10487 Grant Drive, Germantown, WI 53022
Tel 920-215-7012
Cell 920-539-1167
TSC 800-963-4776

From: Paisley, Ken [mailto:KPaisley@sevenson.com]
Sent: Wednesday, July 02, 2014 3:56 PM
To: Barnes, James
Cc: Ramey, Rudy
Subject: RE: Menekaunee Harbor - City of Marinette, WI

Jim,

Got it. Will go through and call next week with any questions. One thing – with testing of soils, is there a landfill requirement for parameters and frequency? From our notes from the site walk, the contractors were being asked to provide this.

There will also be a volume of wood pilings (haven't got a total tonnage yet) that we have to pull and potentially dispose of offsite. Would need to know what testing and how often for these, also. Assuming they'll have to be sized. Will need sizing req'ts.

Kenneth Paisley, CHMM
Technical Services Manager
Sevenson Environmental Services, Inc.

APPENDIX F

Standard Operating Procedures (Sevenson and Robert E. Lee)

Standard Operating Procedures included:

Sevenson – Soil Stockpile Sampling

Sevenson – Lagoon/River Sediment Sampling

Sevenson – Field Documentation

Sevenson – Decontamination

Sevenson – Packing, Storing, and Shipment of Samples

Sevenson – Sample Cooler Shipment

Sevenson – Sediment Sampling Procedures

Sevenson – Chain of Custody Form

Sevenson – Subconsultant UPEA Testing Procedures

Sevenson – Testing Certification

Sevenson – Seametrics Field Calibration Technical Bulletin

Robert E. Lee – Surface Water Sample Collection

Robert E. Lee – Deep Sediment Sample Collection

Robert E. Lee – Chain of Custody Procedures

Robert E. Lee – Equipment Decontamination

Robert E. Lee – Chain of Custody Form

Robert E. Lee – Bathymetric Survey & Calibration

Sevenson Standard Operating Procedures

STANDARD OPERATING PROCEDURES - 120.14

SOIL STOCKPILE SAMPLING

1. Introduction

This attachment presents protocols by which surface soil/sediment stockpile samples will be collected and composited at the site.

II. Equipment

- Stainless steel bucket auger or disposable sample trowel
- Flame Ionization Detector (FID)
- Camera

III. Materials

- Health and safety equipment (as required by the Health and Safety Plan);
- Cleaning equipment;
- Disposable aluminum tray;
- 200-foot tape measure;
- Appropriate sample containers and forms;
- Coolers with ice;
- Field book.

IV. Procedure

The following procedures will be employed to collect surface soil/sediment samples:

1. Don personal protective equipment (as required by the Health and Safety Plan).

2. Identify stockpile sample locations from sample location plan and note locations in field notebook. Provided the safety procedures for exclusion zone entry and employee protective systems consistent with 29 CFR 1926 Subpart P are followed, sampling personnel may sample and collect soil/sediment samples by carefully cutting into the soil with a disposable sample trowel or bucket auger.

3. Transfer the required number of subsamples to a disposable aluminum tray. If VOC analysis is required, place one of the subsamples directly into the appropriate container. Homogenize the remaining subsamples in the aluminum tray and transfer the composite sample to the remaining appropriate containers.

5. Label sample containers and cap in accordance with Sevenson sample handling procedures.

6. Place sample container in a shipping cooler.

7. Discard gloves and disposable sampling equipment in designated location.

8. Handle, pack and ship the samples with appropriate chain-of-custody procedures in accordance with Sevenson SOP 130.20, "Sample Shipment".

9. Record all other appropriate information in the field log book.

10. Decontaminate any non-disposable equipment following standard procedures and wrap in aluminum foil for storage.

V. Disposal Methods

Materials generated during the above activities will be disposed of as per site requirements.

STANDARD OPERATING PROCEDURES - 120.27

LAGOON/RIVER SEDIMENT SAMPLING

Equipment	
Boats and paddles	**Preservative**
Chain-of-custody documents	**Ponar dredge/Push Core/Vibracore Sampler**
Dispatches	**Protective gear**
Garbage bags	**Rubber or hip boots**
Labels	**Sample gloves**
Life jackets	**Sample jars/vials**
Paper towels	**Sample log**

1. Introduction

This attachment presents protocols by which lagoon/river sediment samples will be collected and composited at the site.

II. Equipment

- Ponar Dredge/Push Core/Vibracore Sampler
- Flame Ionization Detector (FID)
- Camera

III. Materials

- Health and safety equipment (as required by the Health and Safety Plan);
- Cleaning equipment;
- Disposable aluminum tray;
- 200-foot tape measure;
- Appropriate sample containers and forms;
- Coolers with ice;
- Field book.

IV. Procedure

The following procedures will be employed to collect surface soil/sediment samples:

1. Don personal protective equipment (as required by the Health and Safety Plan).

2. Identify sample locations from sample location plan and note locations in field notebook. Depending on site conditions and sample requirements, use a ponar drege, puhcore or vibracore sampler to obtain desired sample volumes.

3. Transfer the required number of subsamples to a disposable aluminum tray. If VOC analysis is required, place one of the subsamples directly into the appropriate container. Homogenize the remaining subsamples in the aluminum tray and transfer the composite sample to the remaining appropriate containers.

5. Label sample containers and cap in accordance with in Sevenson sample handling procedures.

6. Place sample container in a shipping cooler.

7. Discard gloves and disposable sampling equipment in designated location.

8. Handle, pack and ship the samples with appropriate chain-of-custody procedures in accordance with Sevenson SOP 130.20, "Sample Shipment".

9. Record all other appropriate information in the field log book.

10. Decontaminate any non-disposable equipment following standard procedures and wrap in aluminum foil for storage.

V. Disposal Methods

Materials generated during the above activities will be disposed of as per site requirements.

TASK SPECIFIC QC CHECKLIST

Work Task: Field Documentation
Sevenson Checklist #: 008

Project Name/Job Number: _____
Inspection Date: _____

Complete this form for each day samples are taken. Answer each question by checking the appropriate column (yes, no, not observed (N/O) or not applicable (N/A). If "no" is checked, provide an explanation of the non-compliance and associated corrective action(s).

		Yes	No	N/A
1.	Was all original field data recorded in black indelible ink?	☐	☐	☐
2.	Were log books filled out properly, accurately recounting the day's events?	☐	☐	☐
3.	Were all field forms completed and information accurately recorded			
	· Field Sampling Forms	☐	☐	☐
	· Chain of Custody Forms	☐	☐	☐
	· Field Log Books	☐	☐	☐
	· FCR/NCR Forms	☐	☐	☐
	List (any) additional forms completed:			
4.	Was field documentation forwarded to Sevenson office for peer/QC review?	☐	☐	☐
5.	Were deficiencies reported to the Field Sampling Manager?	☐	☐	☐

Notes/Comments

QC Inspector Name and Signature _____

Date _____

TASK SPECIFIC QC CHECKLIST

Work Task: Decontamination
Sevenson Checklist #: 009

Project Name/Job Number: _____
Inspection Date: _____

Complete this form for each day samples are taken. Answer each question by checking the appropriate column (yes, no, not observed (N/O) or not applicable (N/A). If "no" is checked, provide an explanation of the non-compliance and associated corrective action(s).

		Yes	No	N/O	N/A
1.	Was all sampling equipment decontaminated properly prior to use and between sample intervals?	☐	☐	☐	☐
2.	Was each decontamination event recorded in the log book?	☐	☐	☐	☐
3.	Was IDW (decontamination water, ppe, etc.) handled properly?	☐	☐	☐	☐
4.	Was the location, type, number and source of containers of IDW recorded in the logbook?	☐	☐	☐	☐
5.	Was SES Technical Services notified If IDW requires offsite disposal?	☐	☐	☐	☐

Notes/Comments

QC Inspector Name and Signature _____

Date _____

TASK SPECIFIC QC CHECKLIST

Work Task: Packing, Storing, and Shipment of Samples
Sevenson Checklist #: 130.13

Project Name/Job Number: _____
Inspection Date: _____

Complete this form for each cooler/shipment inspected. Answer each question by checking the appropriate column (yes, no, not observed (N/O) or not applicable (N/A). If "no" is checked, provide an explanation of the non-compliance and associated corrective action(s).

	Yes	No	N/O	N/A
1. Were the samples handled according to the FSP and QAPP?	☐	☐	☐	☐
2. Did the samples remain on ice or refrigerated (except for sample transfer from coolers or refrigerators) from collection until the cooler was taped for shipment?	☐	☐	☐	☐
3. Where sample containers prepared for shipment (bubble-wrap, Zip-Lock™ bags, etc) per SAP procedures?	☐	☐	☐	☐
4. Was a trip blank (for VOC samples only) and a temperature blank included in each cooler?	☐	☐	☐	☐
5. Was loose ice double Zip-Lock™- bagged prior to placement in cooler?	☐	☐	☐	☐
6. Was ice placed in equal proximity to all sample containers and the temperature blank to ensure samples arrive at lab at 4°C?	☐	☐	☐	☐
7. Were Chain-of-Custody forms filled out accurately and completely, including the project name and number, sampling date and time, analytical parameters, preservatives, size and number of containers for each analytical parameter, and media sampled?	☐	☐	☐	☐
8. Were Chain-of-Custody forms signed and dated by the preparer and the form taped to the inside of the cooler lid?	☐	☐	☐	☐
9. Were signed and dated custody seals properly placed on the cooler and the cooler sealed with strapping tape?	☐	☐	☐	☐
10. Was a shipping label attached to the cooler?	☐	☐	☐	☐
11. Were COCs and shipping tracking labels fax'd to lab?	☐	☐	☐	☐

Notes/Comments

QC Inspector Name and Signature _____

Date _____

TASK SPECIFIC QC CHECKLIST

Work Task: Sample Cooler Shipment
Sevenson Checklist #: SES 130.20

Project Name: _____
Sevenson Job No. _____
QC Inspection Report Date _____

☐ PREPARATORY PHASE

Yes	*No*	*N/A*		*Comment*
☐	☐	☐	Have sample shipment procedures in the SAP been reviewed by all field sampling personnel?	
☐	☐	☐	Are sufficient numbers of clean, hard plastic coolers available onsite to meet current sampling schedule?	
☐	☐	☐	Are sufficient packing supplies (i.e. Ziplock™ plastic bags, packing "peanuts", sealing tape, etc.) available onsite?	
☐	☐	☐	Are laboratory chain-of-custody forms, custody seals, and extra sample container labels available onsite?	
☐	☐	☐	Has a common carrier (Federal Express, UPS, etc.) been selected and have package pickup points and times been identified?	
☐	☐	☐	Will sample coolers be D.O.T. regulated for shipping purposes and are D.O.T. required shipping labels and logs available at the site?	
☐	☐	☐	Have the offsite primary and QA laboratories been contacted to verify anticipated sample collection and shipment schedules?	
☐	☐	☐	Does the primary and QA laboratory have a list of sample shipping site contacts should there be problems or questions?	
☐	☐	☐	Is an ice source available on or near the site?	

☐ INITIAL PHASE OR ☐ FOLLOW-UP PHASE

Yes	*No*	*N/A*		*Comment*
☐	☐	☐	Has a Preparatory Phase meeting been conducted?	
☐	☐	☐	Where sample containers received from the field properly labeled , prepared, and recorded in the field logbook and in the site sample summary log per the SAP?	
☐	☐	☐	Was the sample cooler inspected to verify that it was clean, undamaged, and had no external markings or shipping addresses unrelated to the project?	
☐	☐	☐	Was the cooler inspected to verify that the site name, address, telephone number and contact name was written in indelible ink on the interior of the cooler lid?	
☐	☐	☐	Was the cooler drain plug (if present) securely taped shut on both the interior and exterior sides?	
☐	☐	☐	Was a clean, new plastic garbage bag placed into the cooler as a secondary liner?	

☐	☐	☐	Where all samples, field duplicates, QA splits, and rinse blanks verified by checking sample labels against field logbook entries as chain-of-custody forms were completed?	_____
☐	☐	☐	Were chain-of-custody forms completed per SAP?	_____
☐	☐	☐	Were sample containers placed into separate Ziplock™ bags before being placed in an upright position in the cooler?	_____
☐	☐	☐	Was packing material placed between sample containers to prevent shifting or breakage during shipment?	_____
☐	☐	☐	Was a temperature blank placed in close proximity to sample containers?	_____
☐	☐	☐	If required, was double Ziplock™-bagged ice placed in the cooler in contact with all sample containers?	_____
☐	☐	☐	Was the outer garbage bag sealed with a twist-tie or knot?	_____
☐	☐	☐	Were chain-of-custody forms placed inside a Ziplock™ bag and taped to the inner lid of the cooler?	_____
☐	☐	☐	Was the closed cooler lid checked to verify a proper closure and was fiber-reinforced strapping tape placed around both of its ends at least twice?	_____
☐	☐	☐	Were handling labels and D.O.T. hazard labels (if required) placed on the outside of the cooler?	_____
☐	☐	☐	Were a minimum of two (front and side) completed custody seals placed across the lid opening to verify cooler integrity?	_____
☐	☐	☐	Was an address label with both the “shipped from” and “shipped to” addresses applied to the top of the cooler?	_____
☐	☐	☐	Was the common carrier airbill (or other shipping form) properly completed and attached to the cooler?	_____
☐	☐	☐	Was the “shipper’s copy” of the airbill retained at the site and attached to the DCQCR?	_____
☐	☐	☐	Were all destination laboratories notified of sample shipments?	_____

Comments:_____
_____
_____
_____

Sevenson Project Manager_____
Name Signature Date

QC Reviewer _____
Name Signature Date

CQC Systems Manager _____
Name Signature Date

Sediment Sampling Procedures

1. Don health and safety equipment (as required in the Health and Safety Plan).

2. Use GPS surveying techniques to maneuver the vessel to the target sample location. Secure the vessel in place using spuds, anchors, or tie lines.

3. Measure the total depth of water using a surveyor's rod to the nearest 0.1 foot, and record the water surface elevation with the GPS.

4. Select an appropriate length of 3-inch outside diameter aluminum core tube based on the beneficial reuse material depth.

5. Mount a clean coring tube into the vibracoring device or position with excavator.

6. Lower the core tube attached vertically through the water column tube end first, until it just reaches the top of sediment.

7. Vibrate or push the core to the required depth. Measure and record the depth of core tube penetration into the sediments in the field book.

8. Pull the apparatus upward out of the river or lake bottom, and raise it to the surface, while maintaining the core in a vertical position.

9. Before the bottom of the tube breaks the water surface, place a cap over the bottom end of the tube while still submerged to prevent loss of material from the core tube. The cap will be placed on the core by reaching down into the water from the center of the vessel. Secure the cap in place with duct tape when brought on board the vessel.

10. Water overlying the core tube in the coring apparatus will be allowed to drain prior to removal of the core tube.

11.Estimate the recovered length of the sediment core and note it in the field notebook. The length of the cores recovered in aluminum tubing will be determined directly by lowering a clean aluminum measuring device into the top of the tube. The distance to the top of the sediment in the core tube will be subtracted from the total tube length for a recovered sediment length.

12. After successful core recovery, enter additional information into the field notes: date; time of recovery; sample position; water depth (feet) and water surface elevation; core penetration depth (feet); core recovery depth (feet); and observations, including probing results.

13. Remove the core tube from the device and place a second cap on the top of the core tube. As necessary, and while keeping the core upright, use a pipe cutter (or other appropriate device) to make a horizontal cut in the core tube approximately one inch above the sediment. Secure the cap in place with duct tape. Rinse the outside of the core tube with a small amount of river or lake water.

14. Draw an arrow on the core tube or write “top” on the core tube with permanent marker to mark the top of the core. Label the core with permanent marker indicating station ID, date, and time.

15. Store the core vertically while on the vessel and transport to the processing area.

16. Upon delivery of the cores to the processing area, measure the recovered sediment in the core tube and begin draining the overlying water by drilling a hole just above the sediment interface.

17. Prepare a clean set of disposable aluminum pans for sectioned core segments. Mark each container with location and sample depth.

18. Due to the potential for soft/loose surface sediment, as necessary the upper sections (to one foot depth) may be sectioned using a pipe cutter (or other appropriate device) with the core left in a vertical position. Each of these sections will be carefully transferred into a disposable aluminum pan.

19. For lower sample sections, the core tube will be placed in a horizontal position and electric shears will be utilized to cut the tube lengthwise to expose the full intact core.

20. Physical descriptions of each core will be obtained and changes in stratigraphy noted as they relate to material targeted for beneficial reuse. Characteristics include the general soil type, approximate grain size, presence of observable biota, odor, and color.

21. Remove representative samples based on the designated segmentation scheme.

22. Homogenize samples in a disposable aluminum pan as necessary.

23. Place homogenized sample in appropriate sample containers and cap

Sevenson Environmental Services

CHAIN OF CUSTODY RECORD

PROJECT NO.:	SITE NAME:	NO. OF CON-TAINERS							REMARKS
SAMPLER'S (SIGNATURE):									
STATION NO.	DATE	TIME	COMP	GRAB	STATION LOCATION				

RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED BY (SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED BY (SIGNATURE):
RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED BY (SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED BY (SIGNATURE):
RELINQUISHED BY (SIGNATURE):	DATE / TIME:	RECEIVED FOR LABORATORY BY (SIGNATURE):	DATE / TIME:	REMARKS:	

Distribution: Original accompanies shipment. Copy to coordinator field files.

UPEA ENGINEERS & ARCHITECTS

1701 Dunlap Street, Suite B Marinette, WI 54143
715-732-4188 • 866-682-8418 • Fax: 715-732-4189

August 20, 2014

W. Mark Schmitt
Sevenson Environmental Services, Inc
Menekaunee Harbor Improvements
828 Ogden Street
Marinette, WI 54143
Ph: 715-732-0618 Fax: 715-732-0619
Cell: 716.923.5638

Re: Testing Procedures

Dear Mr. Schmitt

U.P. Engineers and Architects provide the following testing services and follow the practices and procedures per the ASTM listed with each test.

Aggregate Testing

Modified lab Proctor	ASTM- D1557
Standard lab Proctor	ASTM- D698
Particle Size Analysis	ASTM -D422
Stock Pile Sampling	ASTM -D75

Concrete Testing

Concrete Sampling	ASTM-C172
Field Specimens and Curing	ASTM- C31
Concrete Temperature	ASTM- C1064
Concrete Slump Testing	ASTM -C143
Air Content	ASTM -C231
Compressive Strength of Cylinders	ASTM- C39

Density Testing

Density Testing with Troxler Gauge ASTM- D2922

If you have questions, please contact us at 715-732-4188.

Respectfully Submitted,

Jeremy Brunette
Civil Engineer

AMERICAN CONCRETE INSTITUTE

This is to certify that

JEREMY S BRUNETTE

has demonstrated knowledge and ability by successfully completing the ACI Certification requirements and is hereby recognized as an

ACI Concrete Field Testing Technician - Grade I

Certified Date: 05/16/2014 **Expires: 05/16/2019**

Examiner of Record: Mr Steven M Waalkes

ACI Managing Director of Certification

The Authenticity of this certification can be verified at www. ACICertification .org/verify

Technical Bulletin
Field Calibration of Seametric Meters

Seametrics meters leave the factory calibrated and with the K-factor marked on the meter or fitting. However, where the installation is less than ideal (or the original K-factor for the meter has been lost), you may wish to field calibrate your meter to take into account the exact conditions of flow (temperature, pressure, piping configuration, viscosity of liquid) under which it will be used.

Field calibration is accomplished by running a known volume of liquid past the meter, reading the total on your indicator at the end of the procedure, and calculating the number of pulses recorded per volumetric unit of liquid.

Step-by-step instructions for this simple procedure follow:

1. Install the meter and indicator.
2. Locate a bucket, tank, or barrel whose precise volume (in gallons) is known to use as your target container.
3. Reset the total on your indicator to zero, following the instructions in your manual.
4. Reset your indicator K-factor to one (1), following the instructions in your manual.
5. Run the fluid past the meter until the target container is full, without overflow.
6. Read the total on the display of your indicator. This number represents the total number of pulses that were recorded as the known volume of liquid passed the meter.
7. Calculate your K-factor by dividing the total number of pulses by the volume of liquid that flowed past the meter. Usually the K-factor is measured in gallons, but any volumetric unit can be used (for instance, pulses per liter, etc).
8. Reset your indicator with the new, field-calibrated K-factor, following the instructions in your manual.

EXAMPLE: You filled a 5 gallon bucket, and your indicator reads 500. Your field-calibrated K-factor is 500 pulses divided by 5 gallons, or 100 pulses per gallon.

OPTIONAL: If you would like an even more precise K-factor, repeat the above steps at various flow rates typical for your installation and average your K-factor readings.

Seametrics Incorporated • 19026 72nd Avenue South • Kent, Washington 98032 • USA
(P) 253.872.0284 • (F) 253.872.0285 • 1.800.975.8153 • www.seametrics.com

LT-13276-A
3/8/07

Robert E. Lee Standard Operating Procedures

STANDARD OPERATING PROCEDURE 5
Surface Water Sample Collection

Initiator: nll Approved: RLE

1.0 Purpose

The purpose of this standard operating procedure (SOP) is to describe the procedures for the collection of representative surface water samples.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriated site report.

2.0 Equipment

- Sampling and Analysis Plan (SAP)
- Health and Safety Plan (HSP)
- Field book
- Waterproof ink pen and permanent marker
- Any of the following equipment may be used to collect samples:
  - Teflon or stainless steel scoop with a Teflon or stainless steel handle.
  - Stainless steel beaker clamped to a pole
  - Sample bottles (direct fill)
  - Portable peristaltic pump with Teflon tubing
- Appropriate laboratory sample containers
- Sample labels

- Water quality multi-meter (e.g., YSI 556 multi meter) for measuring temperature, specific conductance, pH, dissolved oxygen (DO) and oxidation reduction potential (ORP) or separate individual meters may also be used
- Insulated cooler, chain-of-custody seals, Ziploc® bags
- Decontamination equipment (SOP-3)

3.0 Procedures

1. Immediately before collecting the sample, record the temperature, DO, pH, and specific conductance using the water quality multi-meter or equivalent and following manufacturer's specification by placing in the area of water to be sampled, as needed per project specifications.
2. Submerge the bottle, beaker or scoop and collect a sample. When pouring the water from the beaker or scoop into another bottle container, care should be taken so as not to aerate the sample. Volatile organic compounds (VOCs) will be collected first.
3. A peristaltic pump may also be used to collect the samples, if necessary.
4. The following procedures should be followed when filling VOC containers:

a) Open one vial at a time, set cap in a clean place, and begin collecting the sample.

b) When filling a VOC vial, tip it at a slight angle and allow a steady stream of water to run down its inner wall.

c) Fill the vial until it appears that it is ready to overflow and a positive meniscus forms on the top of the vial.

d) Check that the cap has not been contaminated (splashed) and carefully cap the vial. Place the cap directly over the top and screw down firmly. Do not over tighten.

e) Invert the sample vial and tap gently to check for air bubbles. If an air bubble appears, discard the sample and fill another VOC vial. It is imperative that no entrapped air is in

the sample vial. If air bubbles persist, an unpreserved sample will be collected. This sample shall be noted as such in the field log book and on the chain-of-custody form.

f) Immediately place the sample vial in a protective bubble-wrap bag and place into the cooler.

5. Continue to collect samples in the following order:

a) Non-filtered, non-preserved (e.g., semi-volatile organic compounds [SVOCs], polynuclear aromatic hydrocarbons [PAHs], pesticides, herbicides, PCBs, sulfate, chloride, total chromium VI)

b) Non-filtered, preserved (e.g., total organic carbon [TOC], nitrogen series [ammonia, nitrates, nitrites, etc.], total metals, cyanide)

c) Filtered, non-preserved (e.g., dissolved chromium VI)

d) Filtered , preserved (e.g., dissolved metals except chromium VI)

6. Cap the sample container tightly and place pre-labeled sample container in a bubble-wrap bag. Then place the bubble wrapped sample in a Ziploc® bag. Label Ziploc® bag with the sample identification and project number. Place water sample in an insulated cooler and chill to 4° C.

7. Log all samples in the field book.

8. Decontaminate sampling equipment in accordance with SOP-3.

STANDARD OPERATING PROCEDURE 6
Deep Sediment Sample Collection

Initiator: NH Approved: BLB

1.0 Purpose

The purpose of this standard operating procedure (SOP) is to describe the procedures for the collection of representative deep sediment samples.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriate site report.

2.0 Equipment

- Sampling and Analysis Plan (SAP)
- Health and Safety Plan (HSP)
- Field book
- Waterproof ink pen and permanent marker
- Camera
- Boat
- Ponar Dredge/Vibracore Sampler (or similar devices)
- Photoionization detector (PID)
- Measuring Tape
- Stainless Steel Bowl or Aluminum Foil
- Ziploc® and plastic garbage bags

- Appropriate soil sample containers provided by the laboratory
- EZ Draw plastic syringes
- Sample labels
- Appropriate personal protective equipment (PPE) (i.e, disposable gloves, safety glasses, life vests, rubber boots)
- Insulated cooler, ice, and chain-of-custody seals
- Decontamination equipment (SOP-3)

3.0 Procedures

1. Identify sample locations from SAP or other sample location plan. Based on site conditions and sample requirements, collect sediment for analysis using the appropriate sample device such as a ponar device, vibacore or other similar device.
2. All sediment samples must be handled with disposable gloves. Sample containers will be filled in the order of the most volatile to least volatile in the following order: volatile organic compounds (VOCs), gasoline range organics (GRO), diesel range organics (DRO), semi-volatile organic compounds (SVOCs), polynuclear aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, herbicides, cyanide, total organic carbon (TOC), metals, grain size and/or percent solids analysis.
3. Transfer the sediment from each sample location to a stainless steel bowl or on aluminum foil and fill VOC sample containers for laboratory analysis first as a discrete grab samples (i.e., samples collected for VOC analysis should not be composited), using a clean EZ draw plastic syringe (provided by the laboratory) from a discrete location within the sample interval. Insert the syringe into the soil to obtain 10 grams of soil and continue pushing until the soil column inside the syringe is to the mark labeled "10" and stop drawing soil. Wipe all debris from the outside of the syringe. Immediately remove the cap from a tared, 40-milliliter (mL) vial containing a laboratory pre-measured volume of methanol and insert the soil sample into the vial with the syringe. Be careful to avoid splashing any preservative out

of the sample vial by holding the vial at an angle while slowly extruding the soil core into the vial. Replace cap on vial and secure tightly. Gently swirl the sample vial to completely immerse the soil in the methanol. Soil and methanol ratio is 1:1 (i.e., 10 grams of soil to 10 mL of methanol).

4. Next, collect the GRO soil samples, if specified in the SAP, using the same syringe and preservation procedure as for VOCs, described in Step 2.

5. Collect the DRO soil samples next, if specified in the SAP, using the same plastic syringe from a discrete location within the sample interval. Insert the syringe into the soil to obtain 10 grams of soil and continue pushing until the soil column inside the syringe is to the mark labeled "10" and stop drawing soil. Wipe all debris from the outside of the syringe. Immediately remove the cap from an unpreserved, tared 40-mL vial and insert the soil sample into the vial with the syringe. Replace cap on vial and close tightly.

6. Discard the used syringe in a plastic garbage bag.

7. Homogenize ace remaining sediment within the bowl/aluminum foil by mixing thoroughly with a stainless steel spoon to obtain a homogenous sample representative of the entire sampling interval in preparation for the collection of SVOCs, PAHs, PCBs, pesticides, herbicides, cyanide, total organic carbon (TOC), metals, grain size analysis and/or percent solids, as specified in the SAP.

8. Begin filling the appropriate sample containers with soil using a stainless steel spoon in a manner to minimize the headspace (e.g., the containers must be completely filled) and secure the caps tightly.

9. If required by SAP, place remaining soil in a Ziploc® bag for field screening analysis and follow the soil field screening procedures in SOP-4.

10. Finish labeling sample containers with the time of collection. Place the samples in bubble-wrap bags. Then place the bubble wrapped samples in a Ziploc® bag. Label Ziploc® bags with sample identifications and project number. Place samples in an insulated cooler and chill to 4° C.

11. Log sample collection and location in the field book.

12. Decontaminate sediment sampling equipment (i.e., stainless steel spoons and homogenizing bowls) in accordance with SOP-3. Aluminum foils should be disposed in a plastic garbage bag.

15. Move to next proposed sample location.

STANDARD OPERATING PROCEDURE 9
Chain of Custody Form

Initiator: nll Approved: LLB

1.0 Purpose

The purpose of this standard operating procedure (SOP) is to describe the steps necessary for the proper completion of the laboratory chain of custody form which is used to provide a record of "custody" and to document the transfer of samples from one party to the next (i.e., from the consultant to the laboratory). The chain of custody form identifies each sample container and the individual who has possession of the sample from its origin to its final destination. The chain of custody also describes the sampling point, the date and time the sample was collected, the analytical parameters to be performed on the sample by the laboratory, and the preservation method for the sample. *Note: A sample is defined as being under a person's custody if the following conditions are met:*

- *It is in the person's actual possession*
- *It is in view after being in a person's possession*
- *It is locked up so that on one can tamper with it after having been in a person's physical custody*

2.0 Equipment

- Chain of Custody Form
- Ink pen

3.0 Procedures

1. All applicable portions of the Chain of Custody form must be filled out at the time of sample collection. The Chain of Custody form should be completed by the field personnel responsible for collection of the sample and will follow the sample from the time of collection until the sample has been analyzed by the laboratory.
2. The information included on the Chain of Custody will include, at a minimum, the client name, project name and number, sampler name, unique sample identification, date and time of sample collection, sample matrix (i.e., soil, groundwater, etc.), the number of sample containers associated with each sample point, the type of preservation used, if any, analysis requested for each sample, and contact information for direction of laboratory reports.
3. At the time of transfer of samples to recipient, the sampler will sign the Chain of Custody on the "Relinquished By" field. The date and time must also be included. If the samples are shipped to the laboratory for analysis, the shipping carrier does not sign the Chain of Custody form provided the sample cooler is custody sealed.
4. At the time of sample delivery to the laboratory, the laboratory contact will sign the Chain of Custody on the "Received by Lab" field. Date and time must be included.

STANDARD OPERATING PROCEDURE 3
Equipment Decontamination

Initiator: nll Approved: BLB

1.0 Purpose

The purpose of this standard operating procedure (SOP) is to provide a general guideline for decontamination of sampling and monitoring equipment. Equipment decontamination procedures minimize the potential for cross contamination between sampling points and helps maintain data quality.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriate site report.

2.0 Equipment

- Deionized (DI) Water
- Non-phosphate detergent (e.g., Alconox® or Liqui-Nox®)
- Large plastic pails or tubs for detergent and water
- Spray Bottles for detergent and water
- Scrub brushes
- Plastic bags
- Paper towel

3.0 Procedures

3.1 Non-dedicated Sampling/Monitoring Equipment

1. Wash sampling/monitoring equipment with a non-phosphate detergent and water solution (e.g., Alconox® or Liqui-Nox®).
2. Rinse with DI water and air dry.

3.2 Non-dedicated pumping equipment

1. Wash equipment with a non-phosphate detergent solution and scrub with a scrub brush. For submersible pumps, circulate detergent solution through pump and/or tubing.
2. Thoroughly rinse with DI water.
3. For sampling of organic compounds (i.e., pesticides and herbicides), rinse equipment with an organic desorbing agent (i.e., acetone). For sampling of inorganic compounds (e.g., metals, polycyclic aromatic hydrocarbons, polychlorinated biphenyls), rinse equipment with an inorganic desorbing agent (i.e., dilute hydrochloric acid or nitric acid solution).
4. If an inorganic desorbing agent is used, rinse with potable water.
5. Rinse with DI water and air dry.
6. Place equipment in an inert container, or wrap in clean plastic for storage and transport.

Robert E. Lee & Associates, Inc.
Engineering, Surveying, Environmental Services
1250 Centennial Centre Blvd.
Hobart, WI 54155
920.662.9641 FAX 920.662.9141

To ensure the proper handling of samples, please see the back for instructions.

CHAIN OF CUSTODY RECORD

COC # 201584

Client:

Project Name:

Project Number: BID #:

Environmental Program:
☐ LUST ☐ SDWA ☐ WPDES ☐ RCRA ☐ OTHER _____

Requested Turnaround Time
☐ Normal (10-15 DAYS) ☐ Rush
Date Needed: _____
Rushes accepted only w/prior notification

*Preservation Code
N = Nitric Acid (red)
H = Hydrochloric Acid
M = Methanol
O = Sodium Hydroxide
U = Unpreserved (white)
S = Sulfuric Acid (green)

Sampler:

Sample Type (Matrix)
DW = Drinking Water
GW = Groundwater
WW= Wastewater
Soil, Oil, Sludge, Air, Other:

Analyses Required:
(Note special detection limits or methods)

Filtered ? (Y / N)
Preservation *(Code)
No. Of Containers

Report to:
Company: **Robert E. Lee & Associates**
Address: **1250 Centennial Centre Blvd.**
Hobart, WI 54155
Telephone: **920-662-9641**

Invoice to:
Company: **Robert E. Lee & Associates**
Address: **1250 Centennial Centre Blvd.**
Hobart, WI 54155
Telephone: **920-662-9641**

Sample Name	Date	Time		Comp	Grab	Laboratory Sample I.D.	Remarks:
			A P				
			A P				
			A P				
			A P				
			A P				
			A P				
			A P				
			A P				
			A P				
			A P				
			A P				
			A P				

Relinquished By	Date	Time	Received By	Date	Time
1)		A/P			A/P
2)		A/P			A/P
3)		A/P			A/P

Received by Lab _____ A = AM P = PM

Laboratory Receiving Notes
Temperature of Contents _____ °C
Custody Seal Intact _____
Sample Condition _____
Sample pH _____

Robert E. Lee & Associates, Inc.
Engineering, Surveying, Environmental Services

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PH 920-662-9641 FAX 920-662-9141

MENEKAUNEE HARBOR – MARINETTE, WISCONSIN

BATHYMETRIC SURVEY STANDARD OPERATING PROCEDURES

Equipment

Robert E. Lee & Associates, Inc. will utilize a Trimble R8 GPS receiver in conjunction with a SonarMite MILSpec Echo Sounder. See attached datasheets.

Calibration

The Trimble R8 GPS is factory calibrated. A verification process is done prior to the collection of field date. The predetermined site projection (Wisconsin County Coordinate System – Marinette) is set in to the data collector and at least two published monuments are checked and verified that the equipment meets factory specifications.

The SonarMite MILSpec Echo Sounder is factory calibrated. In order to insure that the unit meets factory specification bottom depths are verified by either lead line or pole methods.

Control & Datum

Robert E. Lee & Associates, Inc. will calibrate the horizontal and vertical control to Station MARINETT as published by the National Geodetic Survey (NGS). We will also verify the onsite benchmark, BM 1 – Corp. of Engineers to ensure the quality of vertical datum. The horizontal and vertical datum's will be referenced to the following:

Horizontal
Wisconsin County Coordinate System (WCCS), Marinette
North American Datum of 1983, Adjustment 1991 (NAD83(91))

Vertical
North American Vertical Datum of 1988 (NAVD88)

Survey Technique

Robert E. Lee & Associates, Inc. will complete the bathymetric survey using the cross-section technique with a single beam echo sounder. Intermittent quality control checks will be done with a lead line. All as described in Chapter 2 of the COE Manual EM-1110-2-1003.

TRIMBLE R8 GNSS SYSTEM

KEY FEATURES

Advanced satellite tracking with **Trimble 360 receiver technology**

Includes Trimble Maxwell 6 chips with **440 channels**

Unmatched GNSS tracking performance

Web user interface and remote configuration

Base and rover communications **options to suit any application**



THE INDUSTRY LEADING TOTAL GNSS SOLUTION

The Trimble® R8 GNSS system has long set the bar for advanced GNSS surveying systems. Through advanced Trimble 360 tracking technology and a comprehensive set of communication options integrated into a flexible system design, this integrated GNSS system delivers industry-leading performance. For surveyors facing demanding RTK applications, the Trimble R8 is an invaluable GNSS partner.

TRIMBLE 360 RECEIVER TECHNOLOGY

Future-proof your investment

Powerful Trimble 360 receiver technology integrated in the Trimble R8 supports signals from all existing and planned GNSS constellations and augmentation systems providing unmatched GNSS tracking performance. With this leading-edge technology, it is now possible for surveyors to expand the reach of their GNSS rovers into areas that were previously too obscured, such as under trees and in dense urban areas.

With two integrated Trimble Maxwell™ 6 chips, the Trimble R8 offers an unparalleled 440 GNSS channels. Also capable of tracking carrier signals from a wide range of satellite systems, including GPS, GLONASS, Galileo, BeiDou (COMPASS), and QZSS, the Trimble R8 provides a robust solution for surveyors.

The CMRx communications protocol in the Trimble R8 provides unprecedented correction compression for optimized bandwidth and full utilization all of the satellites in view, giving you the most reliable positioning performance.

Designed with the future in mind, Trimble 360 technology is optimized to receive future planned signals as the number of available satellites continues to grow. With Trimble 360 technology, the Trimble R8 delivers business confidence with a sound GNSS investment for today and long into the future.

FLEXIBLE SYSTEM DESIGN

The Trimble R8 combines the most comprehensive feature set into an integrated and flexible system design for demanding surveying applications. Connect directly to the controller, receive RTK network corrections, and connect to the Internet via comprehensive communication options. With a built-in transmit/receive UHF radio, the Trimble R8 enables ultimate flexibility for rover or base operation. As a base station, the internal NTRIP caster provides you customized access[1] to base station corrections via the Internet.

Trimble's exclusive Web UI™ eliminates travel requirements for routine monitoring of base station receivers. Now you can assess the health and status of base receivers and perform remote configurations from the office. Likewise, you can download post-processing data through Web UI and save additional trips out to the field.

AN INDUSTRY LEADING FIELD SOLUTION

If you're seeking the industry leading field solution, pair the Trimble R8 GNSS receiver with one of our powerful Trimble controllers, such as the Trimble TSC3, Trimble CU or Trimble Tablet Rugged PC featuring Trimble Access™ field software. These rugged controllers bring the power of the office to the field through an intiutive Windows-based interface.

Trimble Access field software offers numerous features and capabilities to streamline the flow of everyday surveying work. Streamlined workflows such as Roads, Monitoring, Mines, and Tunnels—guide crews through common project types and allows crews to get the job done faster with less distractions. Survey companies can also implement their unique workflows by taking advantage of the customization capabilities available in the Trimble Access Software Development Kit (SDK).

Need to get data back to the office immediately? Benefit from real-time data sharing via Trimble Access Services, now available with any valid Trimble Access maintenance agreement.

Back in the office, seamlessly transfer your field data using Trimble Business Center. Edit, process, and adjust collected data with confidence.

The Trimble R8 GNSS system—the industry leader for GNSS surveying applications.

1 Cellular modem required.

Trimble®

TRIMBLE R8 GNSS SYSTEM

DATASHEET

PERFORMANCE SPECIFICATIONS

Measurements

- Advanced Trimble Maxwell 6 Custom Survey GNSS chips with 440 channels
- Future-proof your investment with Trimble 360 tracking
- High precision multiple correlator for GNSS pseudorange measurements
- Unfiltered, unsmoothed pseudorange measurements data for low noise, low multipath error, low time domain correlation and high dynamic response
- Very low noise GNSS carrier phase measurements with <1 mm precision in a 1 Hz bandwidth
- Signal-to-Noise ratios reported in dB-Hz
- Proven Trimble low elevation tracking technology
- Satellite signals tracked simultaneously:
  - GPS: L1C/A, L1C, L2C, L2E, L5
  - GLONASS: L1C/A, L1P, L2C/A, L2P, L3
  - SBAS: L1C/A, L5 (for SBAS satellites that support L5)
  - Galileo: E1, E5A, E5B
  - BeiDou (COMPASS): B1, B2
- SBAS: QZSS, WAAS, EGNOS, GAGAN
- Positioning rates: 1 Hz, 2 Hz, 5 Hz, 10 Hz, and 20 Hz

POSITIONING PERFORMANCE[1]

Code differential GNSS positioning

Horizontal	0.25 m + 1 ppm RMS
Vertical	0.50 m + 1 ppm RMS
SBAS differential positioning accuracy[2]	typically <5 m 3DRMS

STATIC GNSS SURVEYING

High-precision static

Horizontal	3 mm + 0.1 ppm RMS
Vertical	3.5 mm + 0.4 ppm RMS

Static and FastStatic

Horizontal	3 mm + 0.5 ppm RMS
Vertical	5 mm + 0.5 ppm RMS

POSTPROCESSED KINEMATIC (PPK) GNSS SURVEYING

Horizontal	8 mm + 1 ppm RMS
Vertical	15 mm + 1 ppm RMS

REAL-TIME KINEMATIC SURVEYING

Single Baseline <30 km

Horizontal	8 mm + 1 ppm RMS
Vertical	15 mm + 1 ppm RMS

NETWORK RTK[3]

Horizontal	8 mm + 0.5 ppm RMS
Vertical	15 mm + 0.5 ppm RMS
Initialization time[4]	typically <8 seconds
Initialization reliability[4]	typically >99.9%

1 Precision and reliability may be subject to anomalies due to multipath, obstructions, satellite geometry, and atmospheric conditions. The specifications stated recommend the use of stable mounts in an open sky view, EMI and multipath clean environment, optimal GNSS constellation configurations, along with the use of survey practices that are generally accepted for performing the highest-order surveys for the applicable application including occupation time appropriate for baseline length. Baselines longer than 30 km require precise ephemeris and occupations up to 24 hours may be required to achieve the high precision static specification.
2 Depends on SBAS system performance.
3 Network RTK PPM values are referenced to the closest physical reference station.
4 May be affected by atmospheric conditions, signal multipath, obstructions and satellite geometry. Initialization reliability is continuously monitored to ensure highest quality.
5 Receiver will operate normally to –40 °C, internal batteries are rated to –20 °C, optional internal GSM modem operates to -30 °C.
6 Tracking GPS, GLONASS and SBAS satellites.
7 Varies with temperature and wireless data rate. When using a receiver and internal radio in the transmit mode, it is recommended that an external 6 Ah or higher battery is used.
8 Varies with terrain and operating conditions.
9 Bluetooth type approvals are country specific.

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PN 022543-079M (04/13)

HARDWARE

Physical

Dimensions (W×H)	19 cm × 10.4 cm (7.5 in x 4.1 in), including connectors
Weight	1.52 kg (3.35 lb) with internal battery, internal radio with UHF antenna 3.81 kg (8.40 lb) items above plus range pole, controller, and bracket
Temperature[5]	
Operating	–40 °C to +65 °C (–40 °F to +149 °F)
Storage	–40 °C to +75 °C (–40 °F to +167 °F)
Humidity	100%, condensing
Water/dustproof	IP67 dustproof, protected from temporary immersion to depth of 1 m (3.28 ft)
Shock and vibration	Tested and meets the following environmental standards:
Shock	Non-operating: Designed to survive a 2 m (6.6 ft) pole drop onto concrete. Operating: to 40 G, 10 msec, sawtooth
Vibration	MIL-STD-810F, FIG.514.5C-1

Electrical

- Power 11 V DC to 28 V DC external power input with over-voltage protection on Port 1 (7-pin Lemo)
- Rechargeable, removable 7.4 V, 2.6 Ah Lithium-Ion battery. Power consumption[6] is 3.2 W in RTK rover mode with internal radio and Bluetooth in use.
- Operating times on internal battery:[7]
  - 450 MHz receive only option 5.0 hours
  - 450 MHz receive/transmit option (0.5 W) 2.5 hours
  - Cellular receive option 4.7 hours

Communications and Data Storage

- Serial: 3-wire serial (7-pin Lemo) on Port 1; full RS-232 serial on Port 2 (Dsub 9 pin)
- Radio modem: fully Integrated, fully sealed internal 450 MHz receiver/transmitter option:
  - Transmit power: 0.5 W
  - Range[8]: 3–5 km typical/10 km optimal
- Cellular: fully integrated, sealed internal GSM/GPRS option
- Bluetooth: fully integrated, fully sealed 2.4 GHz communications port (Bluetooth®)[9]
- External communication devices for corrections supported on Serial and Bluetooth ports
- Data storage: 56 MB internal memory, 960 hours of raw observables (approx. 1.4 MB/day), based on recording every 15 sec from an average of 14 satellites

Data formats

- CMR: CMR+, CMRx input and outputs
- RTCM: RTCM 2.1, RTCM 2.3, RTCM 3.0, RTCM 3.1 input and outputs
- Other outputs: 23 NMEA outputs, GSOF, RT17 and RT27 outputs, supports BINEX and smoothed carrier

Web UI

- Offers simple configuration, operation, status and data transfer
- Accessible via Serial and Bluetooth

Supported Trimble Controllers

- Trimble TSC3 controller, Trimble CU controller, Trimble Tablet Rugged PC

Certifications

FCC Part 15 (Class B device), 22, 24, 90; CE Mark; C-Tick; 850/1900 MHz; Class 10 GSM/GPRS module; Bluetooth EPL

Specifications subject to change without notice.

CE C Bluetooth

TRIMBLE AUTHORIZED DISTRIBUTION PARTNER

NORTH AMERICA
Trimble Navigation Limited
10368 Westmoor Dr
Westminster CO 80021
USA

EUROPE
Trimble Germany GmbH
Am Prime Parc 11
65479 Raunheim
GERMANY

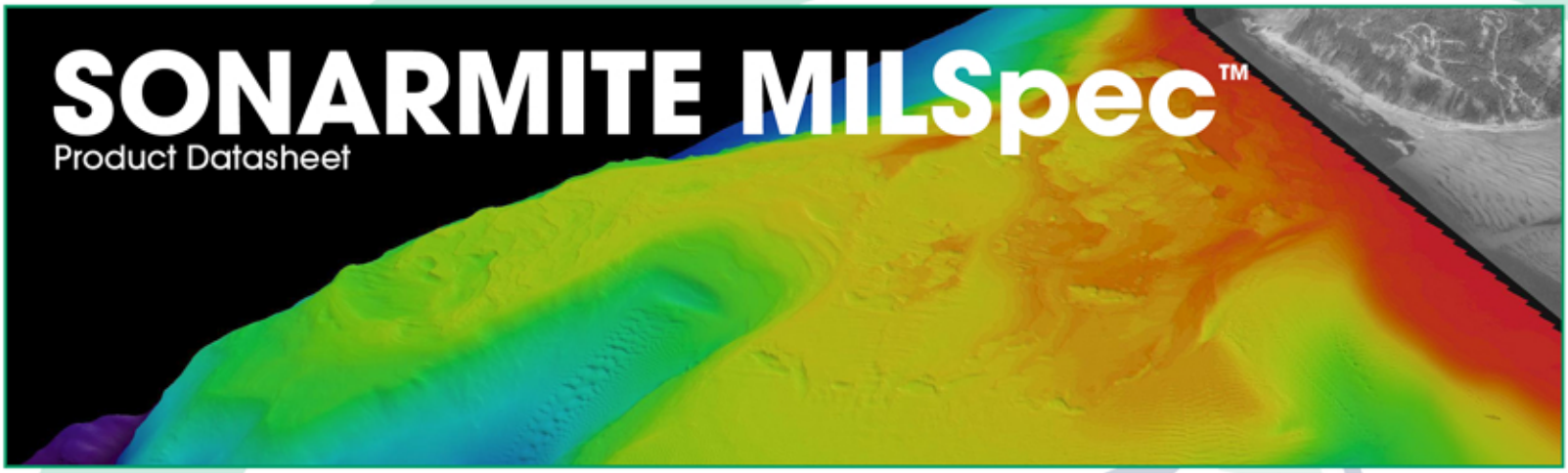
ASIA-PACIFIC
Trimble Navigation
Singapore Pty Limited
80 Marine Parade Road
#22-06, Parkway Parade
Singapore 449269
SINGAPORE

Trimble

Seafloor

SONARMITE MILSpec™

Product Datasheet



ABOUT

The SonarMite MILSpec™ Echo Sounder is result of nearly two years research and development to further extend the boundaries of shallow water hydrographic surveying equipment. The introduction by Ohmex in 1997 of the SonarLite, the world's first truly portable echosounder system, has been a hard act to follow and it remains the portable instrument of choice in many survey companies around the world. The release of the SonarMite instrument marks the next stage introducing a series of equipment designed around the WinSTRUMENT concept using the latest portable computer integrated with new measurement technologies.

FEATURES

- Rugged, field-proven survey grade echosounder
- Bluetooth technology integrated with Windows Pocket PC devices
- Proven 'Smart' transducer design with QA output
- Internal rechargeable battery for all day use
- Easily integrated with other modern software & GPS technology

OPTIONS

- Data collection software
- Heave, Pitch and Roll measurements
- Sound velocimeter
- Portable mounting bracket
- Rugged shipping case
- Extended warranty

SPECS

ECHOSOUNDER

- Frequency: 200-KHz
- Beam width: 4-degrees
- Ping Rate: 6 Hz
- Depth Accuracy: 1cm /0.1% of depth
- Output Formats: NMEA, ASCII
- Range: 0.3m-75m
- I/O: Serial, Bluetooth
- Environmental: IP-65
- Power: Rechargeable 12V battery

PHOTOS





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APPENDIX G

Quality Assurance Manual and Standard Operating Procedures (Pace Analytical)

Standard Operating Procedures included:

Biochemical Oxygen Demand

Total Phosphorus using Block Digestion and Analyzed by SmartChem

Total Cyanide using Micro-Distillation and SmartChem

Measurement of Volatile Solids and Solids in Water

Measurement of pH in Water, Soil, and Waste

The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy

Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010B

The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy – CETAC M-7500

Determination of Hexane Extractable Material (HEM) and Silica Gel Treated-Hexane Extractable Material (SGT-HEM) in Solids

Determination of Total Recoverable Phenolics by Konelab

Determination of Hexane Extractable Material (HEM) and Silica Gel Treated-Hexane Extractable material (SGT-HEM) in Water

DRAFT 8-25-14

QUALITY ASSURANCE MANUAL

Quality Assurance/Quality Control Policies and Procedures

Pace Analytical Services – Green Bay, WI
1241 Bellevue Street Suite 9
Green Bay, WI 54302
920-469-2436

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Chad Rusch, Laboratory Technical Director — Date: 06/03/2014
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Any printed documents in use within a Pace Analytical Services, Inc. laboratory have been reviewed and approved by the persons listed on the cover page. They can only be deemed official if proper signatures are present.

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1.0. INTRODUCTION AND ORGANIZATIONAL STRUCTURE

"Working together to protect our environment and improve our health"
Pace Analytical Services Inc. - Mission Statement

1.1. Introduction to PASI

1.1.1. Pace Analytical Services, Inc. (PASI) is a privately held, full-service analytical testing firm operating a nationwide system of laboratories. PASI offers extensive services beyond standard analytical testing, including: bioassay for aquatic toxicity, air toxics, dioxins and coplanar PCB's by high resolution mass spectroscopy , radiochemical analyses, product testing, pharmaceutical testing, field services and mobile laboratory capabilities. PASI has implemented a consistent Quality System in each of its laboratories and service centers. In addition, the company utilizes an advanced data management system that is highly efficient and allows for flexible data reporting. Together, these systems ensure data reliability and superior on-time performance. This document defines the Quality System and QA/QC protocols.

1.1.2. Our goal is to combine our expertise in laboratory operations with customized solutions to meet the specific needs of our customers.

1.2. Statement of Purpose

1.2.1. To meet the business needs of our customers for high quality, cost-effective analytical measurements and services.

1.3. Quality Policy Statement and Goals of the Quality System

1.3.1. PASI management is committed to maintaining the highest possible standard of service for our customers by following a documented quality system that is fully compliant with the applicable NELAC or TNI standards. The overall objective of this quality system is to provide reliable data of known quality through adherence to rigorous quality assurance policies and quality control procedures as documented in this Quality Assurance Manual.

1.3.2. All personnel within the PASI network are required to be familiar with all facets of the quality system relevant to their position and implement these policies and procedures in their daily work. This daily focus on quality is applied with initial project planning, continued through all field and laboratory activities, and is ultimately included in the final report generation.

1.3.3. PASI management demonstrates its commitment to quality by providing the resources, including facilities, equipment, and personnel to ensure the adherence to these documented policies and procedures and to promote the continuous improvement of the quality system. All PASI personnel must comply with all current applicable state, federal, and industry standards (2003 NELAC Standard, 2009 TNI Standard, etc.), and are required to perform all tests in accordance with stated methods and customer requirements.

1.4. Core Values

1.4.1. **Integrity-** Pace personnel are required to abide by the PASI Code of Ethics and all Pace employees must go through Data Integrity/Ethics training upon initial orientation and as an annual refresher.

1.4.2. **Value Employees-** Pace management views employees as our most important asset and communicates to them the relevance and importance of their activities within their job functions and how they contribute to the achievement of the objectives of the quality management system.

1.4.3. **Know Our Customers-** Pace makes every effort to know our customers and address their sampling and analytical needs. More information on this item can be found in section 2.0.

1.4.4. **Honor Commitments-** Pace labs focus on making solid commitments with regards to quality, capacity, and agreed upon turnaround time to our customers.

1.4.5. **Flexible Response To Demand-** Pace labs are equipped with both the material and personnel resources to enable them to be responsive to the demands of customers when situations or projects need change.

1.4.6. **Pursue Opportunities-** Pace is committed to pursuing opportunities for the growth of the company by constantly exploring markets and areas where we can expand.

1.4.7. **Continuously Improve-** Pace has committed much time and effort into establishing a continuous improvement program where company personnel meet on a regular basis to share ideas in cost reduction, production improvement and standardization in order to develop best practices. This information, as well as company financial and production metrics, are tracked, evaluated, and shared with each Pace facility.

1.5. Code of Ethics

1.5.1. PASI's fundamental ethical principles are as follows:

1.5.1.1. Each PASI employee is responsible for the propriety and consequences of his or her actions;

1.5.1.2. Each PASI employee must conduct all aspects of Company business in an ethical and strictly legal manner, and must obey the laws of the United States and of all localities, states and nations where PASI does business or seeks to do business;

1.5.1.3. Each PASI employee must reflect the highest standards of honesty, integrity and fairness on behalf of the Company with customers, suppliers, the public, and one another.

1.5.1.4. Each PASI employee must recognize and understand that our daily activities in environmental laboratories affect public health as well as the environment and that environmental laboratory analysts are a critical part of the system society depends upon to improve and guard our natural resources:

1.5.2. Strict adherence by each PASI employee to this Code of Ethics and to the Standards of Conduct is essential to the continued vitality of PASI and to continue the pursuit of our common mission to protect our environment and improve our health.

1.5.3. Failure to comply with the Code of Ethics and Standards of Conduct will result in disciplinary action up to and including termination and referral for civil or criminal prosecution

where appropriate. An employee will be notified of an infraction and given an opportunity to explain, as prescribed under current disciplinary procedures.

1.5.4. Any Pace employee can contact corporate management to report an ethical concern by calling the anonymous hotline at 612-607-6431.

1.6. Standards of Conduct

1.6.1. Data Integrity

1.6.1.1. The accuracy and integrity of the analytical results and its supporting documentation produced at PASI are the cornerstones of the company. Lack of data integrity is an assault on our most basic values putting PASI and its employees at grave financial and legal risk and will not be tolerated. Therefore, employees are to accurately prepare and maintain all technical records, scientific notebooks, calculations, and databases. Employees are prohibited from making false entries or misrepresentations of data for any reason.

1.6.1.2. Managerial staff must make every effort to ensure that personnel are free from any undue pressures that may affect the quality or integrity of their work including commercial, financial, over-scheduling, and working condition pressures.

1.6.2. Confidentiality

1.6.2.1. PASI employees must not use or disclose confidential or proprietary information except when in connection with their duties at PASI. This is effective over the course of employment and for an additional period of two years thereafter.

1.6.2.2. Confidential or proprietary information, belonging to either PASI and/or its customers, includes but is not limited to test results, trade secrets, research and development matters, procedures, methods, processes and standards, company-specific techniques and equipment, marketing and customer information, inventions, materials composition, etc.

1.6.3. Conflict of Interest

1.6.3.1. PASI employees must avoid situations that might involve a conflict of interest or could appear questionable to others. The employee must be careful in two general areas:

1.6.3.1.1. Participation in activities that conflict or appear to conflict with the employees' PASI responsibilities.

1.6.3.1.2. Offering or accepting anything that might influence the recipient or cause another person to believe that the recipient may be influenced to behave or in a different manner than he would normally. This includes bribes, gifts, kickbacks, or illegal payments.

1.6.3.2. Employees are not to engage in outside business or economic activity relating to a sale or purchase by the Company. Other problematic activities include service on the Board of Directors of a competing or supplier company, significant ownership in a competing or supplier company, employment for a competing or supplier company, or participation in any outside business during the employee's work hours.

1.6.4. Compliance

1.6.4.1. All employees are required to read, understand, and comply with the various components of the standards listed in this document. As confirmation that they understand their responsibility, each employee is required to sign an acknowledgment form annually that then becomes part of the

employee's permanent record. Employees will be held accountable for complying with the Quality Systems as summarized in the Quality Assurance Manual.

1.7. Laboratory Organization

1.7.1. The PASI Corporate Office centralizes company-wide accounting, business development, financial management, human resources development, information systems, marketing, quality, safety, and training activities. PASI's Director of Quality is responsible for assisting the development, implementation and monitoring of quality programs for the company. See Attachment IIB for the Corporate Organizational structure.

1.7.2. Each laboratory within the system operates with local management, but all labs share common systems and receive support from the Corporate Office.

1.7.3. A Senior General Manager (SGM) oversees all laboratories and service centers in their assigned region. Each laboratory or facility in the company is then directly managed by an SGM, a General Manager (GM), an Assistant General Manager (AGM), or an Operations Manager (OM). Quality Managers (QM) or Senior Quality Managers (SQM) at each laboratory report directly to the highest level of local laboratory management, however named, that routinely makes day-to-day decisions regarding that facility's operations. The QMs and SQMs will also receive guidance and direction from the corporate Director of Quality.

1.7.4. The SGM, GM, AGM or OM, or equivalent functionality in each facility, bears the responsibility for the laboratory operations and serves as the final, local authority in all matters. In the absence of these managers, the SQM/QM serves as the next in command, unless the manager in charge has assigned another designee. He or she assumes the responsibilities of the manager, however named, until the manager is available to resume the duties of their position. In the absence of both the manager and the SQM/QM, management responsibility of the laboratory is passed to the Technical Director, provided such a position is identified, and then to the most senior department manager until the return of the lab manager or SQM/QM. The most senior department manager in charge may include the Client Services Manager or the Administrative Business Manager at the discretion of the SGM/GM/AGM/OM.

1.7.5. A Technical Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director to temporarily perform this function. The laboratory SGM/GM/AGM/OM or SQM/QM has the authority to make this designation in the event the existing Technical Director is unable to do so. If this absence exceeds 35 consecutive calendar days, the primary accrediting authority shall be notified in writing.

1.7.6. The SQM/QM has the responsibility and authority to ensure the Quality System is implemented and followed at all times. In circumstances where a laboratory is not meeting the established level of quality or following the policies set forth in this Quality Assurance Manual, the SQM/QM has the authority to halt laboratory operations should he or she deem such an action necessary. The SQM/QM will immediately communicate the halting of operations to the SGM/GM/AGM/OM and keep them posted on the progress of corrective actions. In the event the SGM/GM/AGM/OM and the SQM/QM are not in agreement as to the need for the suspension, the Chief Operating Officer and Director of Quality will be called in to mediate the situation.

1.7.7. The technical staff of the laboratory is generally organized into the following functional groups:

- Organic Sample Preparation

- Wet Chemistry Analysis
- Metals Analysis
- Volatiles Analysis
- Semi-volatiles Analysis
- Radiochemical Analysis
- Microbiology

1.7.8. Appropriate support groups are present in each laboratory. The actual organizational structure for PASI – Green Bay, WI is listed in Attachment IIA. In the event of a change in SGM/GM/AGM/OM, SQM/QM, or any Technical Director, the laboratory will notify its accrediting authorities and revise the organizational chart in the Quality Assurance Manual (QAM) within 30 days. For changes in Department Managers or Supervisors or other laboratory personnel, no notifications will be sent to the laboratory's accrediting agencies; changes to the organizational chart will be updated during or prior to the annual review process. Changes or additions in these key personnel will also be noted by additional signatures on the QAM, as applicable. In any case, the QAM will remain in effect until the next scheduled revision.

1.8. Laboratory Job Descriptions

1.8.1. Senior General Manager

- Oversees all functions of all the operations within their designated region;
- Oversees the development of local GMs/AGMs/OMs within their designated region;
- Oversees and authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Oversees the preparation of budgets and staffing plans for all operations within their designated region;
- Ensures compliance with all applicable state, federal and industry standards;
- Works closely with Regional Sales Management.

1.8.2. General Manager

- Oversees all functions of their assigned operations;
- Authorizes personnel development including staffing, recruiting, training, workload scheduling, employee retention and motivation;
- Prepares budgets and staffing plans;
- Monitors the Quality Systems of the laboratory and advises the SQM/QM accordingly;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.4. Quality Manager

- Responsible for implementing, maintaining and improving the quality system while functioning independently from laboratory operations. Reports directly to the highest level of local laboratory facility management, however named, that routinely makes day-to-day decisions regarding laboratory operations, but receives direction and assistance from the Corporate Director of Quality. They may also report to a Senior Quality Manager within the same facility;

- Ensures that communication takes place at all levels within the lab regarding the effectiveness of the quality system and that all personnel understand their contributions to the quality system;
- Monitors Quality Assurance/Quality Control activities to ensure that the laboratory achieves established standards of quality (as set forth by the Corporate Quality office). The Quality Manager is responsible for reporting the lab's level of compliance to these standards to the Corporate Director of Quality on a quarterly basis;
- Maintains records of quality control data and evaluates data quality;
- Conducts periodic internal audits and coordinates external audits performed by regulatory agencies or customer representatives;
- Reviews and maintains records of proficiency testing results;
- Maintains the document control system;
- Assists in development and implementation of appropriate training programs;
- Provides technical support to laboratory operations regarding methodology and project QA/QC requirements;
- Maintains certifications from federal and state programs;
- Ensures compliance with all applicable state, federal and industry standards;
- Maintains the laboratory training records, including those in the Learning Management System (LMS), and evaluates the effectiveness of training;
- Monitors correctives actions;
- Maintains the currency of the Quality Manual.

1.8.5. Quality Analyst

- Assists the SQM/QM in the performance of quality department responsibilities as delegated by the SQM/QM;
- Assists in monitoring QA/QC data;
- Assists in internal audits;
- Assists in maintaining training records;
- Assists in maintaining the document control system;

1.8.6. Technical Director

- Monitors the standards of performance in quality assurance and quality control data;
- Monitors the validity of analyses performed and data generated;
- Reviews tenders, contracts and QAPPs to ensure the laboratory can meet the data quality objectives for any given project;
- Serves as the manager of the laboratory in the absence of the SGM/GM/AGM/OM and SQM/QM;
- Provides technical guidance in the review, development, and validation of new methodologies.

1.8.7. Administrative Business Manager

- Responsible for financial and administrative management for the entire facility;
- Provides input relative to tactical and strategic planning activities;
- Organizes financial information so that the facility is run as a fiscally responsible business;

- Works with staff to confirm that appropriate processes are put in place to track revenues and expenses;
- Provide ongoing financial information to the SGM/GM/AGM/OM and the management team so they can better manage their business;
- Utilizes historical information and trends to accurately forecast future financial positions;
- Works with management to ensure that key measurements are put in place to be utilized for trend analysis—this will include personnel and supply expenses, and key revenue and expense ratios;
- Works with SGM/GM/AGM/OM to develop accurate budget and track on an ongoing basis;
- Works with entire management team to submit complete and justified capital budget requests and to balance requests across departments;
- Works with project management team and administrative support staff to ensure timely and accurate invoicing.

1.8.8. Client Services Manager

- Oversees all the day to day activities of the Client Services Department which includes Project Management and, possibly, Sample Control;
- Responsible for staffing and all personnel management related issues for Client Services;
- Serves as the primary senior consultant to customers on all project related issues such as set up, initiation, execution and closure;
- Performs or is capable of performing all duties listed for that of Project Manager.

1.8.9. Project Manager

- Coordinates daily activities including taking orders, reporting data and analytical results;
- Serves as the primary technical and administrative liaison between customers and PASI;
- Communicates with operations staff to update and set project priorities;
- Provides results to customers in the requested format (verbal, hardcopy, electronic, etc.);
- Works with customers, laboratory staff, and other appropriate PASI staff to develop project statements of work or resolve problems of data quality;
- Responsible for solicitation of work requests, assisting with proposal preparation and project initiation with customers and maintain customer records;
- Mediation of project schedules and scope of work through communication with internal resources and management;
- Responsible for preparing routine and non-routine quotations, reports and technical papers;
- Interfaces between customers and management personnel to achieve customer satisfaction;
- Manages large-scale complex projects;
- Supervises less experienced project managers and provide guidance on management of complex projects;
- Arranges bottle orders and shipment of sample kits to customers;
- Verifies login information relative to project requirements and field sample Chains-of-Custody.

1.8.10. Project Coordinator

- Responsible for preparation of project specifications and provides technical/project support;

- Coordinates project needs with other department sections and assists with proposal preparation;
- Prepares routine proposals and invoicing;
- Responsible for scanning, copying, assembling and binding final reports;
- Other duties include filing, maintaining forms, process outgoing mail, maintaining training database and data entry.

1.8.11. Department Manager/Supervisor

- Oversees the day-to-day production and quality activities of their assigned department;
- Ensures that quality assurance and quality control criteria of analytical methods and projects are satisfied;
- Assesses data quality and takes corrective action when necessary;
- Approves and releases technical and data management reports;
- Ensures compliance with all applicable state, federal and industry standards.

1.8.12. Group Supervisor/Leader

- Trains analysts in laboratory operations and analytical procedures;
- Organizes and schedules analyses with consideration for sample holding times;
- Implements data verification procedures by assigning data verification duties to appropriate personnel;
- Evaluates instrument performance and supervises instrument calibration and preventive maintenance programs;
- Reports non-compliance situations to laboratory management including the SQM/QM.

1.8.13. Laboratory Analyst

- Performs detailed preparation and analysis of samples according to published methods and laboratory procedures;
- Processes and evaluates raw data obtained from preparation and analysis steps;
- Generates final results from raw data, performing primary review against method criteria;
- Monitors quality control data associated with analysis and preparation. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks;
- Reports data in LIMS, authorizing for release pending secondary approval;
- Conducts routine and non-routine maintenance of equipment as required;
- Performs or is capable of performing all duties associated with that of Laboratory Technician.

1.8.14. Laboratory Technician

- Prepares standards and reagents according to published methods or in house procedures;
- Performs preparation and analytical steps for basic laboratory methods;
- Works under the direction of a Laboratory Analyst on complex methodologies;
- Assists Laboratory Analysts on preparation, analytical or data reduction steps for complex methodologies;

- Monitors quality control data as required or directed. This includes examination of raw data such as chromatograms as well as an inspection of reduced data, calibration curves, and laboratory notebooks.

1.8.15. Sample Management Personnel

- Signs for incoming samples and verifies the data entered on the Chain of custody forms;
- Enters the sample information into the Laboratory Information Management System (LIMS) for tracking and reporting;
- Stages samples according to EPA requirements;
- Assists Project Managers and Coordinators in filling bottle orders and sample shipments.

1.8.16. Systems Administrator or Systems Manager

- Assists with the creation and maintenance of electronic data deliverables (EDDs);
- Coordinates the installation and use of all hardware, software and operating systems;
- Performs troubleshooting on all aforementioned systems;
- Trains new and existing users on systems and system upgrades;
- Maintains all system security passwords;
- Maintains the electronic backups of all computer systems.

1.8.17. Safety/Chemical Hygiene Officer

- Maintains the laboratory Chemical Hygiene Plan;
- Plans and implements safety policies and procedures;
- Maintains safety records;
- Organizes and/or performs safety training;
- Performs safety inspections and provides corrective/preventative actions;
- Assists personnel with safety issues.

1.8.18. Program Director/Hazardous Waste Coordinator (or otherwise named)

- Evaluates waste streams and helps to select appropriate waste transportation and disposal companies;
- Maintains complete records of waste disposal including waste manifests and state reports;
- Assists in training personnel on waste-related issues such as waste handling and storage, waste container labeling, proper satellite accumulation, secondary containment, etc.;
- Conducts a weekly inspection of the waste storage areas of the laboratory.

1.9. Training and Orientation

1.9.1. Training for Pace employees is managed through a web-based Learning Management System. After a new employee has been instructed in matters of human resources, they are given instructional materials for the LMS and a password for access.

1.9.2. A new hire training checklist is provided to the new employee that lists training items for the employee to work through either independently on LMS or with their supervisor or trainer. The training items that can be completed independently include:

- Reading through applicable Standard Operating Procedures;
- Reviewing the Quality Manual and Chemical Hygiene Plan;
- Core training modules such as quality control indicators, basic laboratory skills, etc.;
- Quality Systems training including traceability of measurements, method calibration, calibration verification, accuracy, precision and uncertainty of measurements, corrective actions, documentation, and root cause analysis;
- Data Integrity/Ethics training.

1.9.3. The new employee's Department Supervisor provides the employee with a basic understanding of the role of the laboratory within the structure of PASI and the basic elements of that individual's position. Supervised training uses the following techniques:

- Hands-on training
- Training checklists/worksheets
- Lectures and training sessions
- Method-specific training
- Conferences and seminars
- Short courses
- Specialized training by instrument manufacturers
- Proficiency testing programs.
- On-line courses

1.9.4. Group Supervisors/Leaders are responsible for providing documentation of training and proficiency for each employee under their supervision. The employee's training file indicates what procedures an analyst or a technician is capable of performing, either independently or with supervision. The files also include documentation of continuing capability, which are fully detailed in Section 3.4. Training documentation files for each person are maintained by the Quality Office either in hardcopy format or within the LMS.

1.9.5. All procedures and training records are maintained and available for review during laboratory audits. These procedures are reviewed/updated periodically by laboratory management. Additional information can be found in SOP S-ALL-Q-020 **Training and Employee Orientation** or its equivalent revision or replacement.

1.10. Data Integrity System

1.10.1. The data integrity system at PASI provides assurances to management that a highly ethical approach is being applied to all planning, training and implementation of methods. Data integrity is crucial to the success of our company and Pace Analytical is committed to creating and maintaining a culture of quality throughout the organization. To accomplish this goal, PASI has implemented a data integrity system that encompasses the following four requirements:

1.10.1.1. A data integrity training program: standardized training is given to each new employee and a yearly refresher is presented to all employees. Key topics addressed by this training include:

1.10.1.1.1. Need for honesty and transparency in analytical reporting
1.10.1.1.2. Process for reporting data integrity issues
1.10.1.1.3. Specific examples of unethical behavior and improper practices
1.10.1.1.4. Documentation of non-conforming data that is still useful to the data user
1.10.1.1.5. Consequences and punishments for unethical behavior

1.10.1.1.6. Examples of monitoring devices used by management to review data and systems

1.10.1.2. Signed data integrity documentation for all employees: this includes a written quiz following the Ethics training session and written agreement to abide by the Code of Ethics and Standards of Conduct explained in the employee manual.

1.10.1.3. In-depth, periodic monitoring of data integrity including peer data review and validation, internal raw data audits, proficiency testing studies, etc.

1.10.1.4. Documentation of any review or investigation into possible data integrity infractions. This documentation, including any disciplinary actions involved, corrective actions taken, and notifications to customers must be retained for a minimum of five years.

1.10.2. PASI management makes every effort to ensure that personnel are free from any undue pressures that affect the quality of their work including commercial, financial, over scheduling, and working condition pressures.

1.10.3. Corporate management also provides all PASI facilities a mechanism for confidential reporting of data integrity issues that ensures confidentiality and a receptive environment in which all employees are comfortable discussing items of ethical concern. The anonymous message line is monitored by the Corporate Director of Quality who will ensure that all concerns are evaluated and, where necessary, brought to the attention of executive management and investigated. Any Pace employee can contact corporate management to report an ethical concern by calling the anonymous hotline at 612-607-6431.

1.11. Laboratory Safety

1.11.1. It is the policy of PASI to make safety and health an integral part of daily operations and to ensure that all employees are provided with safe working conditions, personal protective equipment, and requisite training to do their work without injury. Each employee is responsible for his/her own safety as well as those working in the immediate area by complying with established company rules and procedures. These rules and procedures as well as a more detailed description of the employees' responsibilities are contained in the corporate Safety Manual and Chemical Hygiene Plan.

1.12. Security and Confidentiality

1.12.1. Security is maintained by controlled access to laboratory buildings. Exterior doors to laboratory buildings remain either locked or continuously monitored by PASI staff. Keyless door lock combinations and computer access codes/logins are changed periodically. Posted signs direct visitors to the reception office and mark all other areas as off limits to unauthorized personnel. All visitors, including PASI staff from other facilities, must sign the Visitor's Logbook maintained by the receptionist. A staff member will accompany them during the duration of their stay on the premises unless the SGM/GM/AGM/OM, SQM/QM, or Technical Director specify otherwise. In this instance, the staff member will escort the visitor back to the reception area at the end of his/her visit where he/she signs out. The last staff member to leave their department for the day must ensure that all outside access points to that area are secure.

1.12.2. Additional security is provided where necessary, (e.g., specific secure areas for sample, data, and customer report storage), as requested by customers, or cases where national security is of concern. These areas are lockable within the facilities, or are securely offsite. Access is limited to specific individuals or their designees. Security of sample storage areas is the responsibility of the Sample

Custodian. Security of samples and data during analysis and data reduction is the responsibility of Group Supervisors. Security of customer report archives is the responsibility of the Client Services Manager. These secure areas are locked whenever these individuals or their designees are not present in the facility.

1.12.3. Access to designated laboratory sample storage locations is limited to authorized personnel only. Provisions for lock and key access are provided. No samples are to be removed without proper authorization. If requested by customer or contract, samples are not to be removed from secure storage areas without filling out an associated internal chain of custody.

1.12.4. Standard business practices of confidentiality are applied to all documents and information regarding customer analyses. Specific protocols for handling confidential documents are described in PASI SOPs. Additional protocols for sample identification by internal laboratory identification numbers only are implemented as required under contract specific Quality Assurance Project Plans (QAPPs).

1.12.5. All information pertaining to a particular customer, including national security concerns will remain confidential. Data will be released to outside agencies only with written authorization from the customer or where federal or state law requires the company to do so.

1.13. Communications

1.13.1. Management within each lab bears the responsibility of ensuring that appropriate communication processes are established and that communication takes place regarding the effectiveness of the management/quality system. These communication processes may include email, regular staff meetings, senior management meetings, etc.

1.13.2. Corporate management bears the responsibility of ensuring that appropriate communication processes are established within the network of facilities and that communication takes place at a company-wide level regarding the effectiveness of the management/quality systems of all Pace facilities. These communication processes may include email, quarterly continuous improvement conference calls for all lab departments, and annual continuous improvement meetings for all department supervisors, quality managers, client services managers, and other support positions.

2.0. SAMPLE CUSTODY

2.1. Sampling Support

2.1.1. Each individual PASI laboratory provides shipping containers, properly preserved sample containers, custody documents, and field quality control samples to support field-sampling events. Guidelines for sample container types, preservatives, and holding times for a variety of methods are listed in Attachment VIII. Note that all analyses listed are not necessarily performed at all PASI laboratories and there may be additional laboratory analyses performed that are not included in these tables. Customers are encouraged to contact their local Pace Project Manager for questions or clarifications regarding sample handling. PASI – Green Bay, WI may provide pick-up and delivery services to their customers when needed.

2.2. Field Services

2.2.1. Pace Analytical has a large Field Services Division which is based in their Minneapolis facility as well as limited field service capabilities in some of our other facilities. Field Services provides comprehensive nationwide service offerings including:

- Stack Testing
- Ambient Air
- CEM Certification Testing
- Air Quality Monitoring
- Onsite Analytical Services- FTIR and GC
- Real-time Process Diagnostic/Optimization Testing
- Wastewater, Groundwater and Drinking Water Monitoring
- Storm Water and Surface Water Monitoring
- Soil and Waste Sampling
- Mobile Laboratory Services

2.2.2. Field Services operates under the PASI Corporate Quality System, with applicable and necessary provisions to address the activities, methods, and goals specific to Field Services. All procedures and methods used by Field Services are documented in Standard Operating Procedures and Procedure Manuals.

2.3. Project Initiation

2.3.1. Prior to accepting new work, the laboratory reviews its performance capability. The laboratory confirms that sufficient personnel, equipment capacity, analytical method capability, etc., are available to complete the required work. Customer needs, certification requirements, and data quality objectives are defined and the appropriate sampling and analysis plan is developed to meet the project requirements by project managers or sales representatives. Members of the management staff review current instrument capacity, personnel availability and training, analytical procedures capability, and projected sample load. Management then informs the sales and client services personnel whether or not the laboratory can accept the new project via written correspondence, email, and/or daily operations meetings.

2.3.2. The laboratory maintains records of all such reviews, including discussions with customers. Routine analytical project documentation of quotes, notes, dates, initials, and/or recordings is maintained in a project folder by project management. Conditions for new and more complex contracts are determined by the SGM/GM/AGM/OM and sales representatives. Quality Management is consulted on technical requirements and operations staff provides input on volume capacities. Evidence of these reviews is maintained in the form of awarded Request for Proposals (RFPs), signed quotes or contracts, and a Customer Relationship Management (CRM) database. If a review identifies a potential mismatch between customer requirements and laboratory capabilities and/or capacities, Pace will specify its level of commitment by listing these exceptions to the requirements within the RFP, quote or contract.

2.3.3. Additional information regarding specific procedures for reviewing new work requests can be found in SOP S-GB-C-012 **Review of Analytical Requests** or its equivalent revision or replacement.

2.4. Chain of Custody

2.4.1. A chain of custody (COC) provides the legal documentation of samples from time of collection to completion of analysis. PASI has implemented Standard Operating Procedures to ensure that sample custody traceability and responsibility objectives are achieved for every project.

2.4.2. Field personnel or client representatives must complete a chain of custody for all samples that are received by the laboratory. The importance of completeness of COCs is stressed to the samplers and is critical to efficient sample receipt and to insure the requested methods are used to analyze the correct samples.

2.4.3. If sample shipments are not accompanied by the correct documentation, the Sample Receiving department notifies a Project Manager. The Project Manager then obtains the correct documentation/information from the customer in order for analysis of samples to proceed.

2.4.4. The sampler is responsible for providing the following information on the chain of custody form:

- Customer project name
- Project location or number
- Field sample number/identification
- Date and time sampled
- Sample matrix
- Preservative
- Requested analyses
- Sampler signature
- Relinquishing signature
- Date and time relinquished
- Sampler remarks as needed
- Custody Seal Number if present
- Regulatory Program Designation
- The state where the samples were collected to ensure all applicable state requirements are met
- Turnaround time requested
- Purchase order number

2.4.5. The COC is filled out completely and legibly with indelible ink. Errors are corrected by drawing a single line through the initial entry and initialing and dating the change. All transfers of samples are recorded on the chain of custody in the "relinquished" and "received by" sections. All information except signatures is printed.

2.4.6. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.5. Sample Acceptance Policy

2.5.1. In accordance with regulatory guidelines, PASI complies with the following sample acceptance policy for all samples received.

2.5.2. If the samples do not meet the sample receipt acceptance criteria outlined below, the laboratory is required to document all non-compliances, contact the customer, and either reject the samples or fully document any decisions to proceed with analyses of samples which do not meet the criteria. Any results reported from samples not meeting these criteria are appropriately qualified on the final report.

2.5.3. All samples must:

- Have unique customer identification that is clearly marked with indelible ink on durable waterproof labels affixed to the sample containers that match the chain of custody.
- Have clear documentation on the chain of custody related to the location of the sampling site with the time and date of sample collection.
- Have the sampler's name and signature.
- Have all requested analyses clearly designated on the COC.
- Have clear documentation of any special analytical or data reporting requirements.
- Be in appropriate sample containers with clear documentation of the preservatives used.
- Be correctly preserved unless the method allows for laboratory preservation.
- Be received within holding time. Any samples with hold times that are exceeded will not be processed without prior customer approval.
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without customer approval.
- Be received within appropriate temperature ranges - not frozen but ≤6°C [(See Note 1)], unless program requirements or customer contractual obligations mandate otherwise [(see Note 2)]. The cooler temperature is recorded directly on the COC and the SCUR. Samples that are delivered to the laboratory immediately after collection are considered acceptable if there is evidence that the chilling process has been started. For example, by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the customer will be notified. The analysis will NOT proceed unless otherwise directed by the customer. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the customer is contacted to avoid missing the hold time. Data associated with any deviations from the above sample acceptance policy requirements will be appropriately qualified.

Note 1: Temperature will be read and recorded based on the precision of the measuring device. For example, temperatures obtained from a thermometer graduated to 0.1°C will be read and recorded to

±0.1°C. Measurements obtained from a thermometer graduate to 0.5°C will be read to ±0.5°C. Measurements read at the specified precision are not to be rounded down to meet the ≤6°C limit

Note 2: Some microbiology methods allow sample receipt temperatures of up to 10°C. Consult the specific method for microbiology samples received above 6°C prior to initiating corrective action for out of temperature preservation conditions.

Note 3: Biological Tissue Samples must be received on ice at ≤6°C.

2.5.4. Upon sample receipt, the following items are also checked and recorded:

- Presence of custody seals or tapes on the shipping containers;
- Sample condition: Intact, broken/leaking, bubbles in VOA samples;
- Sample holding time;
- Sample pH and residual chlorine when required;
- Appropriate containers.

2.5.5. Samples for drinking water analysis that are improperly preserved, or are received past holding time, are rejected at the time of receipt, with the exception of VOA samples that are tested for pH at the time of analysis.

2.5.6. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.6. Sample Log-in

2.6.1. After sample inspection, all sample information on the chain of custody is entered into the Laboratory Information Management System (LIMS). This permanent record documents receipt of all sample containers including:

- Customer name and contact
- Customer number
- Pace Analytical project number
- Pace Analytical Project Manager
- Sample descriptions
- Due dates
- List of analyses requested
- Date and time of laboratory receipt
- Field ID code
- Date and time of collection
- Any comments resulting from inspection for sample rejection

2.6.2. All samples received are logged into the LIMS within one working day of receipt. Sample login may be delayed due to customer clarification of analysis needed, corrective actions for sample receipt non-conformance, or other unusual circumstances. If the time collected for any sample is unspecified and Pace is unable to obtain this information from the customer, the laboratory will use 08:00 as the time sampled. All hold times will be based on this sampling time and qualified accordingly if exceeded.

2.6.3. The Laboratory Information Management System (Epic Pro) automatically generates a unique identification number for each sample created in the system. The LIMS sample number follows the general convention of BB-XXXXX-YYY. The BB represents the laboratory identification within Pace's laboratory network. The 5 digit "X" number represents the project number followed by a 3 digit sample number. The project number is a sequential number that is assigned as a new project is created. The sample number corresponds to the number of samples submitted by the client. In addition to the unique sample ID, there is a sample container ID that consists of the sample number, the container type (ex. BP1U), and bottle 1 of Y, where Y represents the total number of containers of that particular type. Together the sample LIMs number and sample container ID number create a unique barcode encryption that can be linked to the sample analysis requested by the client. This unique identification number is placed on the sample container as a durable label and becomes the link between the laboratory's sample management system and the customer's field identification; it will be a permanent reference number for all future interactions.

2.6.4. Current division codes are noted below. These division codes are used primarily for accounting purposes and LIMS sample identifications. For smaller facilities, these codes may be used primarily for accounting purposes. More division codes may be added, revised or removed without updating this document.

00 = Corporate
10 = Minnesota/Montana/Virginia MN
12 = Virginia/Duluth MN
20 = New Orleans/Puerto Rico
30 = Pittsburgh
35 = Florida/South Florida
36 = South Florida (accounting only)
40 = Green Bay
50 = Indianapolis/Columbus
51 = Columbus (accounting only)
55/56 = Pace Energy Labs
60 = Kansas
65 = New York (Schenectady)
70 = Long Island
75 = Dallas
92 = Carolinas

2.6.5. Sample labels are printed from the LIMS and affixed to each sample container.

2.6.6. Samples with hold times that are near expiration date/time may be sent directly to the laboratory for analysis at the discretion of the Project Manager and/or SGM/GM/AGM/OM.

2.6.7. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.7. Sample Storage

2.7.1. Storage Conditions

2.7.1.1. Samples are stored away from all standards, reagents, or other potential sources of contamination. Samples are stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

2.7.1.2. Storage blanks, consisting of two 40mL aliquots of reagent water, are stored with volatile samples and are used to measure cross-contamination acquired during storage. If applicable, laboratories must have documented procedures and criteria for evaluating storage blanks, appropriate to the types of samples being stored.

2.7.1.3. Additional information can be found in most current revision or replacement of SOP: S-GB-Q-028 **Monitoring Temperature Controlled Units.**

2.7.2. **Temperature Monitoring**

2.7.2.1. Samples are taken to the appropriate storage location immediately after sample receipt and check-in procedures are completed. All sample storage areas are located in limited access areas and are monitored to ensure sample integrity.

2.7.2.2. The temperature of each refrigerated storage area is maintained at ≤6°C (but above freezing) unless state or program requirements differ. The temperature of each freezer storage area is maintained at <-10°C unless state or program requirements differ. The temperature of each storage area is checked and documented each day of use (each calendar day). If the temperature falls outside the acceptable limits, the following corrective actions are taken and appropriately documented:

- The temperature is rechecked after two hours to verify temperature exceedance. Corrective action is initiated and documented if necessary.
- The SQM/QM and/or laboratory management are notified if the problem persists.
- The samples are relocated to a proper environment if the temperature cannot be maintained after corrective actions are implemented.
- The affected customers are notified.
- Documentation is provided on analytical report.

Additional information can be found in S-GB-Q-028 **Monitoring Temperature Controlled Units.**

2.7.3. **Hazardous Materials**

2.7.3.1. Pure product or potentially heavily contaminated samples must be tagged as "hazardous" or "lab pack" and stored separately from other samples.

2.7.4. **Foreign/Quarantined Soils**

2.7.4.1. Depending on the soil disposal practices of the laboratory, foreign soils and soils from USDA regulated areas are adequately segregated to enable proper sample disposal. The USDA requires these samples to be incinerated or sterilized by an approved treatment procedure. Additional information regarding USDA regulations and sample handling can be found in applicable local laboratory SOPs.

2.7.4.2. Additional information on sample storage can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement and in S-GB-W-001 **Waste Handling and Management**.

2.8. Sample Protection

2.8.1. PASI laboratory facilities are operated under controlled access protocols to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted at all times.

2.8.2. Samples are removed from storage areas by designated personnel and returned to the storage areas, if necessary, immediately after the required sample quantity has been taken.

2.8.3. Upon customer request, additional and more rigorous chain of custody protocols for samples and data can be implemented. For example, some projects may require internal chain-of-custody protocols.

2.8.4. Additional information can be found in S-GB-C-010 **Sample Management** or its equivalent revision or replacement.

2.9. Subcontracting Analytical Services

2.9.1. Every effort is made to perform all analyses for PASI customers within the laboratory that receives the samples. When subcontracting to a laboratory other than the receiving laboratory, whether inside or outside the PASI network, becomes necessary, a preliminary verbal communication with that laboratory is undertaken. Customers are notified in writing of the laboratory's intention to subcontract any portion of the testing to another laboratory. Work performed under specific protocols may involve special considerations.

2.9.2. Prior to subcontracting samples to a laboratory outside Pace Analytical, the potential sub-contract laboratory will be pre-qualified by verifying that the subcontractor meets the following criteria:

- All certifications required for the proposed subcontract are in effect,
- Sufficient professional liability and other required insurance coverage is in effect, and
- Is not involved in legal action by any federal, state, or local government agency for data integrity issues and has not been convicted in such investigation at any time during the past 5 years.

2.9.3. The contact and preliminary arrangements are made between the PASI Project Manager and the appropriate subcontract laboratory personnel. The specific terms of the subcontract laboratory agreement include:

- Method of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Laboratory certification requirement
- Price per analysis
- Turn-around time requirements

2.9.4. Chain-of-custody forms are generated for samples requiring subcontracting to other laboratories. Sample receiving personnel re-package the samples for shipment, create a transfer chain of custody form and record the following information:

- Pace Analytical Laboratory Number
- Matrix
- Requested analysis
- Special instructions regarding turnaround, required detection or reporting limits, or any unusual information known about the samples or analytical procedure.
- Signature in "Relinquished By"

2.9.5. All subcontracted sample data reports are sent to the PASI Project Manager. Pace will provide a copy of the subcontractor's report to the client when requested.

2.9.6. Any Pace Analytical work sent to other labs within the PASI network is handled as subcontracted work and all final reports are labeled clearly with the name of the laboratory performing

the work. Any non-TNI work is clearly identified. PASI will not be responsible for analytical data if the subcontract laboratory was designated by the customer.

2.9.7. Additional information can be found in S-GB-C-009 **Subcontracting Samples** or its equivalent revision or replacement.

2.10. Sample Retention and Disposal

2.10.1. Samples, extracts, digestates, and leachates must be retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

2.10.2. Unused portions of samples are retained by each laboratory based on program or customer requirements for sample retention and storage. The minimum sample retention time is 45 days from receipt of the samples. Samples requiring thermal preservation may be stored at ambient temperature when the hold time is expired, the report has been delivered, and/or allowed by the customer, program, or contract. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

2.10.3. After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposition of hazardous samples is to return the excess sample to the customer. If it is not feasible to return samples, or the customer requires PASI to dispose of excess samples, proper arrangements will be made for disposal by an approved contractor.

2.10.4. Additional information can be found in S-GB-W-001 **Waste Handling and Management** and S-GB-C-010 **Sample Management** or their equivalent revisions or replacements.

3.0. ANALYTICAL CAPABILITIES

3.1. Analytical Method Sources

3.1.1. PASI laboratories are capable of analyzing a full range of environmental samples from a variety of matrices, including air, surface water, wastewater, groundwater, soil, sediment, biota, and other waste products. The latest valid editions of methodologies are applied from regulatory and professional sources including EPA, ASTM, USGS, NIOSH, Standard Methods, and State Agencies. Section 11 is a representative listing of general analytical protocol references. PASI discloses in writing to its customers and regulatory agencies any instances in which modified methods are being used in the analysis of samples.

3.1.2. In the event of a customer-specific need, instrumentation constraint or regulatory requirement, PASI laboratories reserve the right to use valid versions of methods that may not be the most recent edition available.

3.2. Analytical Method Documentation

3.2.1. The primary form of PASI laboratory documentation of analytical methods is the Standard Operating Procedure (SOP). SOPs contain pertinent information as to what steps are required by an analyst to successfully perform a procedure. The required contents for the SOPs are specified in the company-wide SOP for Preparation of SOPs (S-ALL-Q-001).

3.2.2. The SOPs may be supplemented by other training materials that further detail how methods are specifically performed. This training material will undergo periodic, documented review along with the other Quality System documentation.

3.3. Analytical Method Validation and Instrument Validation

3.3.1. In some situations, PASI develops and validates methodologies that may be more applicable to a specific problem or objective. When non-standard methods are required for specific projects or analytes of interest, or when the laboratory develops or modifies a method, the laboratory validates the method prior to applying it to customer samples. Method validity is established by meeting criteria for precision and accuracy as established by the data quality objectives specified by the end user of the data. The laboratory records the validation procedure, the results obtained and a statement as to the usability of the method. The minimum requirements for method validation include evaluation of sensitivity, quantitation, precision, bias, and selectivity of each analyte of interest.

3.4. Demonstration of Capability (DOC)

3.4.1. Analysts complete an initial demonstration of capability (IDOC) study prior to performing a method or when there is a change in instrument type, personnel, or test method, or at any time that a method has not been performed by the laboratory or analyst in a 12-month period. The mean recovery and standard deviation of each analyte, taken from 4 replicates of a quality control standard is calculated and compared to method criteria (if available) or established laboratory criteria for evaluation of acceptance. Each laboratory maintains copies of all demonstrations of capability, including those that fail acceptance criteria and corresponding raw data for future reference and must

document the acceptance criteria prior to the analysis of the DOC. Demonstrations of capability are verified on an annual basis.

3.4.2. For Continuing Demonstrations of Capability, the laboratories may use Performance Testing (PT) samples in lieu of the 4-replicate approach listed above. For methods or procedures that do not lend themselves to the "4-replicate" approach, the demonstration of capability requirements will be specified in the applicable SOP. Drinking Water DOCs must be done at or below the MCL.

3.4.3. Additional information can be found in SOP S-ALL-Q-020 **Training and Employee Orientation** or its equivalent revision or replacement.

3.5. Regulatory and Method Compliance

3.5.1. PASI understands that expectations of our customers commonly include the assumption that laboratory data will satisfy specific regulatory requirements. Therefore PASI attempts to ascertain, prior to beginning a project, what applicable regulatory jurisdiction, agency, or protocols apply to that project. This information is also required on the chain of custody submitted with samples.

3.5.2. PASI makes every effort to detect regulatory or project plan inconsistencies, based upon information from the customer, and communicate them immediately to the customer in order to aid in the decision making process. PASI will not be liable if the customer chooses not to follow PASI recommendations.

3.5.3. It is PASI policy to disclose in a forthright manner any detected noncompliance affecting the usability of data produced by our laboratories. The laboratory will notify customers within 30 days of fully characterizing the nature of the nonconformance, the scope of the nonconformance and the impact it may have on data usability.

4.0. QUALITY CONTROL PROCEDURES

Quality control data is analyzed and where they are found to be outside pre-defined criteria, planned action is taken to correct the problem in order to prevent incorrect results from being reported. Quality control samples are to be processed in the same manner as client samples.

4.1. Method Blank

4.1.1. A method blank is used to evaluate contamination in the preparation/analysis system and is processed through all preparation and analytical steps with its associated samples.

4.1.2. A method blank is processed at a minimum frequency of one per preparation batch. In the case of a method that has no separate preparation step, a method blank is processed with no more than 20 samples of a specific matrix performed by the same analyst, using the same method, standards, and reagents.

4.1.3. The method blank consists of a matrix similar to the associated samples that is known to be free of analytes of interest. Method blanks are not applicable for certain analyses, such as pH, conductivity, flash point and temperature.

4.1.4. Each method blank is evaluated for contamination. The source of any contamination is investigated and documented corrective action is taken when the concentration of any target analyte is detected above the reporting limit and is greater than 1/10 of the amount of that analyte found in any associated sample. Some labs, due to client requirements, etc., may have to evaluate their method blanks down to ½ the reporting limit or down to the method detection limit as opposed to the reporting limit itself. Corrective actions for blank contamination may include the re-preparation and re-analysis of all samples (where possible) and quality control samples. Data qualifiers must be applied to results that are considered affected by contamination in a method blank.

4.1.5. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.2. Laboratory Control Sample

4.2.1. The Laboratory Control Sample (LCS) is used to evaluate the performance of the entire analytical system including preparation and analysis.

4.2.2. An LCS is processed at a minimum frequency of one per preparation batch. In the case of a method that has no separate preparation step, an LCS will be processed with no more than 20 samples of a specific matrix performed by the same analyst, using the same method, standards, and reagents.

4.2.3. The LCS consists of a matrix similar to the associated samples that is known to be free of the analytes of interest that is then spiked with known concentrations of target analytes.

4.2.4. The LCS contains **all** analytes specified by a specific method or by the customer or regulatory agency, which may include full list of target compounds, with certain exceptions. These exceptions may include analyzing only specific Aroclors when PCB analysis is requested or not spiking with all EPA Appendix IX compounds when a full Appendix IX list of compounds is requested. However, the lab must ensure that all target components in its scope of accreditation are included in the spike

mixture for the LCS over a two (2) year period. In the absence of specified components, the laboratory will spike the LCS with the following compounds:

- For multi-peak analytes (e.g. PCBs, technical chlordane, toxaphene), a representative standard will be processed.
- For methods with long lists of analytes, a representative number of target analytes may be chosen. The following criteria is used to determine the number of LCS compounds used:
  - For methods with 1-10 target compounds, the laboratory will spike with all compounds;
  - For methods with 11-20 target compounds, the laboratory will spike with at least 10 compounds or 80%, whichever is greater;
  - For methods with greater than 20 compounds, the laboratory will spike with at least 16 compounds.

4.2.5. The LCS is evaluated against the method default or laboratory-derived acceptance criteria. For those methods that require laboratory-derived limits, method default control limits may be used until the laboratory has a minimum of 20, but preferably greater than 30, data points from which to derive internal acceptance criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Any associated sample containing an 'out-of-control' compound must either be re-analyzed with a successful LCS or reported with the appropriate data qualifier. When the acceptance criteria for the LCS are exceeded high, and there are associated samples that are non-detects, then those non-detects can be reported with data qualifiers, or when the acceptance criteria are exceeded low, those associated sample results may be reported if they exceed the maximum regulatory limit/decision level with data qualifiers.

4.2.6. For LCSs containing a large number of analytes, it is statistically likely that a few recoveries will be outside of control limits. This does not necessarily mean that the system is out of control, and therefore no corrective action would be necessary (except for proper documentation). TNI has allowed for a minimum number of marginal exceedances, defined as recoveries that are beyond the LCS control limits (3X the standard deviation) but less than the marginal exceedance limits (4X the standard deviation). The number of allowable exceedances depends on the number of compounds in the LCS. If more analyte recoveries exceed the LCS control limits than is allowed (see below) or if any one analyte exceeds the marginal exceedance limits, then the LCS is considered non-compliant and corrective actions are necessary. The number of allowable exceedances is as follows:

- >90 analytes in the LCS- 5 analytes
- 71-90 analytes in the LCS- 4 analytes
- 51-70 analytes in the LCS- 3 analytes
- 31-50 analytes in the LCS- 2 analytes
- 11-30 analytes in the LCS- 1 analyte
- <11 analytes in the LCS- no analytes allowed out)

4.2.7. A matrix spike (MS) can be used in place of a non-compliant LCS in a batch as long as the MS passes the LCS acceptance criteria (this is a TNI allowance). When this happens, full documentation must be made available to the data user. If this is not allowed by a customer or regulatory body, the associated samples must be rerun with a compliant LCS (if possible) or reported with appropriate data qualifiers.

4.2.8. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

4.3.1. A matrix spike (MS) is used to determine the effect of the sample matrix on compound recovery for a particular method. The information from these spikes is sample or matrix specific and is not used to determine the acceptance of an entire batch unless the MS is actually used as the LCS.

4.3.2. A **Matrix Spike/Matrix Spike Duplicate** (MS/MSD) set is processed at a frequency specified in a particular method or as determined by a specific customer request. This frequency will be specified in the applicable method SOP or customer QAPP. In the absence of such requirements, an MS/MSD set is routinely analyzed once per every 20 samples per matrix per method.

4.3.3. The MS and MSD consist of the sample matrix that is then spiked with known concentrations of target analytes. Laboratory personnel spike customer samples that are specifically designated as MS/MSD samples or, when no designated samples are present in a batch, randomly select samples to spike that have adequate sample volume or weight. Spiked samples are prepared and analyzed in the same manner as the original samples and are selected from different customers if possible.

4.3.4. The MS and MSD contain all analytes specified by a specific method or by the customer or regulatory agency. In the absence of specified components, the laboratory will spike the MS/MSD with the same number of compounds as previously discussed in the LCS section. However, the lab must ensure that all targeted components in its scope of accreditation are included in the spike mixture for the MS/MSD over a two (2) year period.

4.3.5. The MS and MSD are evaluated against the method or laboratory derived criteria. Any compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Batch acceptance, however, is based on method blank and LCS performance, not on MS/MSD recoveries. The spike recoveries give the data user a better understanding of the final results based on their site specific information.

4.3.6. A matrix spike and sample duplicate will be performed instead of a matrix spike and matrix spike duplicate when specified by the customer or method.

4.3.7. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.4. Sample Duplicate

4.4.1. A sample duplicate is a second portion of sample that is prepared and analyzed in the laboratory along with the first portion. It is used to measure the precision associated with preparation and analysis. A sample duplicate is processed at a frequency specified by the particular method or as determined by a specific customer.

4.4.2. The sample and duplicate are evaluated against the method or laboratory derived criteria for relative percent difference (RPD). Any duplicate that is outside of these limits is considered to be 'out of control' and must be qualified appropriately.

4.4.3. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.5. Surrogates

4.5.1. Surrogates are compounds that reflect the chemistry of target analytes and are typically added to samples for organic analyses to monitor the effect of the sample matrix on compound recovery.

4.5.2. Surrogates are added to each customer sample (for applicable organics), method blank, LCS, MS, and calibration standard prior to extraction or analysis. The surrogates are evaluated against the method or laboratory derived acceptance criteria or against project-specific acceptance criteria specified by the client, if applicable. Any surrogate compound that is outside of these limits is considered to be 'out of control' and must be qualified appropriately. Samples with surrogate failures are typically re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error. An exception to this would be samples that have high surrogate values but no reportable hits for target compounds. These samples would be reported, with a qualifier, because the implied high bias would not affect the final results. For methods with multiple surrogates, documentation regarding acceptance and associated compounds will be found in the individual method SOPs.

4.5.3. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.6. Internal Standards

4.6.1. Internal Standards are method-specific analytes added to every standard, method blank, laboratory control sample, matrix spike, matrix spike duplicate, sample, and calibration standard at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. At a minimum, the laboratory will follow method specific guidelines for the treatment of internal standard recoveries as they are related to the reporting of data.

4.6.2. Deviations made from this policy must be approved by the SQM/QM prior to release of the data.

4.7. Field Blanks

4.7.1. Field blanks are blanks prepared at the sampling site in order to monitor for contamination that may be present in the environment where samples are collected. These field quality control samples are often referenced as field blanks, rinsate blanks, or equipment blanks. The laboratory analyzes these field blanks as normal samples and informs the customer if there are any target compounds detected above the reporting limits.

4.8. Trip Blanks

4.8.1. Trip blanks are blanks that originate from the laboratory as part of the sampling event and are used to monitor for contamination of samples during transport. These blanks accompany the empty sample containers to the field and then accompany the collected samples back to the laboratory. These blanks are routinely analyzed for volatile methods where ambient background contamination is likely to occur.

4.9. Limit of Detection (LOD)

4.9.1. PASI laboratories are required to use a documented procedure to determine a limit of detection for each analyte of concern in each matrix reported. All sample processing steps of the preparation and analytical methods are included in this determination including any clean ups. For any test that does not have a valid LOD, sample results below the limit of quantitation (LOQ) cannot be reported.

4.9.2. The LOD is initially established for the compounds of interest for each method in a clean matrix with no target analytes present and no interferences at a concentration that would impact the results. The LOD is then determined every time there is a change in the test method that affects how the test is performed or when there has been a change in the instrument that affects the sensitivity. If required by customer, method or accreditation body, the LOD will be re-established annually for all applicable methods.

4.9.3. Unless otherwise noted, the method used by PASI laboratories to determine LODs is based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. Where required by regulatory program or customer, the above referenced procedure will be followed.

4.9.4. Where specifically stated in the published method, LODs or MDLs will be performed at the listed frequency.

4.9.5. The validity of the LOD must be shown by detection (a value above zero) of the analytes in a QC sample in each quality system matrix. The QC sample must contain the analyte at no more than 3X the LOD for a single analyte test and 4X the LOD for multiple analyte tests. This verification must be performed on each instrument used for sample analysis and reporting of data. The validity of the LOD must be verified as part of the LOD determination process. This verification must be done prior to the use of the LOD for sample analysis.

4.9.6. An LOD study is not required for any analyte for which spiking solutions or quality control samples are not available such as temperature.

4.9.7. The LOD, if required, shall be verified annually for each quality system matrix, technology and analyte. In lieu of performing full LOD (MDL) studies annually, the laboratory can verify the LOD (MDL) on an annual basis, providing this verification is fully documented and does not contradict other customer or program requirements that the laboratory must follow. The requirements of this verification are:

- The spike concentration of the verification must be no more than 3X times the LOD for single analyte tests and 4X the LOD for multiple analyte tests.
- The laboratory must verify the LOD on each instrument used for the reporting of sample data.
- The laboratory must be able to identify all target analytes in the verification standard (distinguishable from noise).

4.9.8. Additional information can be found in SOP S-GB-Q-020 **Determination of LOD and LOQ** or its equivalent revision or replacement.

4.10. Limit of Quantitation (LOQ)

4.10.1. A limit of quantitation (LOQ) for every analyte of concern must be determined. For PASI laboratories, this LOQ is referred to as the RL, or Reporting Limit. This RL is based on the lowest

calibration standard concentration that is used in each initial calibration. Results below this level are not allowed to be reported without qualification since the results would not be substantiated by a calibration standard. For methods with a determined LOD, results can be reported out below the LOQ but above the LOD if they are properly qualified (e.g., J flag).

4.10.2. The LOQ must be higher than the LOD.

4.10.3. To verify the LOQ, the laboratory will prepare a sample in the same matrix used for the LCS. The sample will be spiked with each target analyte at a concentration equivalent to the RL or 2X the RL. This sample must undergo the routine sample preparation procedure including any routine sample cleanup steps. The sample is then analyzed and the recovery of each target analyte determined. The recovery for each target analyte must meet the laboratories current control limits for an LCS. The annual LOQ verification is not required if the LOD was determined or verified annually on that instrument.

4.10.4. Additional information can be found in SOP S-GB-Q-020 **Determination of LOD and LOQ** or its equivalent revision or replacement.

4.11. Estimate of Analytical Uncertainty

4.11.1. PASI laboratories can provide an estimation of uncertainty for results generated by the laboratory. The estimate quantifies the error associated with any given result at a 95% confidence interval. This estimate does not include bias that may be associated with sampling. The laboratory has a procedure in place for making this estimation. In the absence of a regulatory or customer-specific procedure, PASI laboratories base this estimation on the recovery data obtained from the Laboratory Control Spikes. The uncertainty is a function of the standard deviation of the recoveries multiplied by the appropriate Student's t Factor at 95% confidence. Additional information pertaining to the estimation of uncertainty and the exact manner in which it is derived are contained in the SOP S-GB-Q-010 **Estimation of Measurement Uncertainty** or its equivalent revision or replacement.

4.11.2. The measurement of uncertainty is provided only on request by the customer, as required by specification or regulation and when the result is used to determine conformance within a specification limit.

4.12. Proficiency Testing (PT) Studies

4.12.1. PASI laboratories participate in the TNI defined proficiency testing program. PT samples are obtained from NIST approved providers and analyzed and reported at a minimum of two times per year for the relevant fields of testing per matrix.

4.12.2. The laboratory initiates an investigation whenever PT results are deemed 'unacceptable' by the PT provider. All findings and corrective actions taken are reported to the SQM/QM or their designee. A corrective action plan is initiated and this report is sent to the appropriate state accreditation agencies for their review. Additional PTs will be analyzed and reported as needed for certification purposes.

4.12.3. PT samples are treated as typical customer samples, utilizing the same staff, methods, equipment, facilities, and frequency of analysis. PT samples are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

4.12.4. Comparison of analytical results with anyone participating in the same PT study is prohibited prior to the close of the study.

4.12.5. Additional information can be found in SOP S-GB-Q-021 **Proficiency Testing Program** or its equivalent revision or replacement.

4.13. Rounding and Significant Figures

4.13.1. In general, the PASI laboratories report data to no more than three significant digits. Therefore, all measurements made in the analytical process must reflect this level of precision. In the event that a parameter that contributes to the final result has less than three significant figures of precision, the final result must be reported with no more significant figures than that of the parameter in question. The rounding rules listed below are descriptive of the LIMS and not necessarily of any supporting program such as Excel.

4.13.2. Data is compared to the reporting limits and MDLs to determine if qualifiers are needed before the rounding step occurs.

4.13.3. **Rounding:** PASI-Green Bay follows the odd / even guidelines for rounding numbers:

- If the figure following the one to be retained is less than five, that figure is dropped and the retained ones are not changed (with three significant figures, 2.544 is rounded to 2.54).
- If the figure following the ones to be retained is greater than five, that figure is dropped and the last retained one is rounded up (with three significant figures, 2.546 is rounded to 2.55).
- If the figure following the ones to be retained is five and if there are no figures other than zeros beyond that five, then the five is dropped and the last figure retained is unchanged if it is even and rounded up if it is odd (with three significant figures, 2.525 is rounded to 2.52 and 2.535 is rounded to 2.54).

4.13.4. **Significant Digits**

4.13.4.1. PASI-Green Bay follows the following convention for reporting to a specified number of significant figures. Unless specified by federal, state, or local requirements or on specific request by a customer, the laboratory reports:

Values > 10 – Reported to 3 significant digits
Values ≤ 10 – Reported to 2 significant digits

4.14. Retention Time Windows

4.14.1. When chromatographic conditions are changed, retention times and analytical separations are often affected. As a result, two critical aspects of any chromatographic method are the determination and verification of retention times and analyte separation. Retention time windows must be established for the identification of target analytes. The retention times of all target analytes in all calibration verification standards must fall within the retention time windows. If an analyte falls outside the retention time window in an ICV or CCV, new absolute retention time windows must be calculated, unless instrument maintenance fixes the problem. When a new column is installed, a new retention time window study must be performed.

4.14.2. One process for the production of retention time windows: Make 3 injections of all single component or multi-component analytes over a 72-hour period. Record the retention time in minutes for each analyte and surrogate to 3 decimal places. Calculate the mean and standard deviation of the three absolute retention times for each target analyte and surrogate. For multi-component analytes, choose 3-5 major peaks and calculate the mean and standard deviation for each of the peaks. If the standard deviation of the retention times of a target analyte is 0.000, the lab may use a default standard deviation of 0.01. The width of the retention time window for each analyte and surrogate and major peak in a multi-component analyte is defined as +/- 3 times the standard deviation of the mean absolute retention time established during that 72-hour period or 0.03 minutes, whichever is greater.

4.14.3. The center of the retention time window is established for each analyte and surrogate by using the absolute retention times from the CCV at the beginning of the analytical shift. For samples run with an initial calibration, use the retention time of the mid-point standard of the initial calibration curve.

4.14.4. For more information, please reference the local facility's analytical SOPs.

5.0. DOCUMENT MANAGEMENT AND CHANGE CONTROL

5.1. Document Management

5.1.1. Additional information can be found in SOP S-GB-Q-029 **Document Control and Management** or its equivalent revision or replacement. Information on Pace's policy for electronic signatures can also be found in this SOP.

5.1.2. Pace Analytical Services, Inc. has an established procedure for managing documents that are part of the quality system. The list of managed documents includes, but is not limited to, Standard Operating Procedures (both technical and non-technical), Quality Assurance Manuals, quality policy statements, training documents, work-processing documents, charts, posters, memoranda, notices, forms, software, and any other procedures, tables, plans, etc. that have a direct bearing on the quality system (including applicable data records and non-technical documents).

5.1.3. A master list of all managed documents is maintained at each facility identifying the current revision status and distribution of the controlled documents. This establishes that there are no invalid or obsolete documents in use in the facility. All documents are reviewed periodically and revised if necessary. Obsolete documents are systematically discarded or archived for audit or knowledge preservation purposes.

5.1.4. Each managed document is uniquely identified to include the date of issue, the revision identification, page numbers, the total number of pages and the issuing authorities. For complete information on document numbering, refer to SOP S-ALL-Q-003 **Document Numbering**.

5.1.5. SOPs, specifically, are available to all laboratory staff via the Learning Management System (LMS) which is a secure repository that is accessed through an internet portal. As a local alternative to the hard copy system of controlled documents, secured electronic copies of controlled documents may be maintained on the laboratory's local server. These document files must be read-only for all personnel except the Quality Department and system administrator. Other requirements for this system are as follows:

- Electronic documents must be readily accessible to all facility employees.
- Electronic documents (i.e. pdf's) must be locked from printing. All hardcopy SOPs must be obtained from the Quality Department.

5.1.6. **Quality Assurance Manual (QAM):** The Quality Assurance Manual is the company-wide document that describes all aspects of the quality system for PASI. The base QAM template is distributed by the Corporate Quality Department to each of the SQMs/QMs. The local management personnel modify the necessary and permissible sections of the base template and submit those modifications to the Corporate Director of Quality for review. Once approved and signed by both the CEO and the Director of Quality; the SGM/GM/AGM/OM, the SQM/QM, and any Technical Directors sign the Quality Assurance Manual. Each SQM/QM is then in charge of distribution to employees, external customers or regulatory agencies and maintaining a distribution list of controlled document copies. The Quality Assurance Manual template is reviewed on an annual basis by all of the PASI SQMs/QMs and revised accordingly by the Director of Quality.

5.1.7. **Standard Operating Procedures (SOPs)**

5.1.7.1. SOPs fall into two categories: company-wide documents and facility specific documents. Company-wide SOPs start with the prefix S-ALL- and local SOPs start with the individual facility prefix.

5.1.7.2. The purpose of the company-wide SOPs is to establish policies and procedure that are common and applicable to all PASI facilities. Company-wide SOPs are document-controlled by the corporate quality office and signed copies are distributed to all of the SQMs/QMs. The local management personnel sign the company-wide SOPs. The SQM/QM is then in charge of distribution to employees, external customers, or regulatory agencies and maintaining a distribution list of controlled document copies.

5.1.7.3. Local PASI facilities are responsible for developing facility-specific SOPs applicable to their respective facility. The local facility develops these facility-specific SOPs based on the corporate-wide SOP template. This template is written to incorporate a set of minimum method requirements and PASI best practice requirements. The local facilities may add to or modify the corporate-wide SOP template provided there are no contradictions to the minimum method or best practice requirements. Facility-specific SOPs are controlled by the applicable SQM/QM according to the corporate document management policies.

5.1.7.4. SOPs are reviewed every two years at a minimum although a more frequent review may be required by some state or federal agencies or customers. If no revisions are made based on this review, documentation of the review itself is made by the addition of new signatures on the cover page. If revisions are made, documentation of the revisions is made in the revisions section of each SOP and a new revision number is applied to the SOP. This provides a historical record of all revisions.

5.1.7.5. All copies of superseded SOPs are removed from general use and the original copy of each SOP is archived for audit or knowledge preservation purposes. This ensures that all PASI employees use the most current version of each SOP and provides the SQM/QM with a historical record of each SOP.

5.1.7.6. Additional information can be found in SOP S-GB-Q-017 **Preparation of SOPs** or its equivalent revision or replacement.

5.2. Document Change Control

5.2.1. Changes to managed documents are reviewed and approved in the same manner as the original review. Any revision to a document requires the approval of the applicable signatories. After revisions are approved, a revision number is assigned and the previous version of the document is officially retired. Copies may be kept for audit or knowledge preservation purposes.

5.2.2. All controlled copies of the previous document are replaced with controlled copies of the revised document and the superseded copies are destroyed or archived. All affected personnel are advised that there has been a revision and any necessary training is scheduled.

5.3. Management of Change

5.3.1. The process for documenting necessary changes within the laboratory network are not typically handled using the corrective or preventive action system as outlined in section 9.0. Management of Change is a proactive approach to dealing with change to minimize the potential negative impact of systematic change in the laboratory and to ensure that each change has a positive desired outcome. This process will primarily be used for the implementation of large scale projects and information system changes as a means to apply consistent systems or procedures within the laboratory network. The request for change is submitted by the initiator and subsequently assigned to an individual or team for development and planning. The final completion of the process culminates in final approval and verification that the procedure was effectively implemented. Additional information can be found in SOP S-GB-Q-016 **Management of Change** or its equivalent revision or replacement.

6.0. EQUIPMENT AND MEASUREMENT TRACEABILITY

Each PASI facility is equipped with sufficient instrumentation and support equipment to perform the relevant analytical testing or field procedures performed by each facility. Support equipment includes chemical standards, thermometers, balances, disposable and mechanical pipettes, etc. This section details some of the procedures necessary to maintain traceability and to perform proper calibration of instrumentation and support equipment. See Attachment III for a list of equipment currently used at the Green Bay PASI facility.

6.1. Standards and Traceability

6.1.1. Each PASI facility retains all pertinent information for standards, reagents, and chemicals to assure traceability to a national standard. This includes documentation of purchase, receipt, preparation, and use.

6.1.2. Upon receipt, all purchased standard reference materials are recorded into a standard logbook or database and assigned a unique identification number. The entries include the facility's unique identification number, the chemical name, manufacturer name, manufacturer's identification numbers, receipt date, and expiration date. Vendor's certificates of analysis for all standards, reagents, or chemicals are retained for future reference.

6.1.3. Subsequent preparations of intermediate or working solutions are also documented in a standard logbook or database. These entries include the stock standard name and lot number, the manufacturer name, the solvents used for preparation, the solvent lot number and manufacturer, the preparation steps, preparation date, expiration dates, preparer's initials, and a unique PASI identification number. This number is used in any applicable sample preparation or analysis logbook so the standard can be traced back to the standard preparation record. This process ensures traceability back to the national standard.

6.1.4. All prepared standard or reagent containers include the PASI identification number, the standard or chemical name, the date of preparation, the date of expiration, the concentration with units, and the preparer's initials. This ensures traceability back to the standard preparation logbook or database.

6.1.5. For containers that are too small to accommodate labels that list all of the above information associated with a standard, the minimum required information will be PASI standard ID, concentration, and expiration date. This assures that no standard will be used past its assigned expiration date.

6.1.6. If a second source standard is required to verify an existing calibration or spiking standard, this standard must be obtained from a different manufacturer or from a different lot unless client specific QAPP requirements state otherwise.

6.1.7. Additional information concerning standards and reagent traceability can be found in the SOP S-GB-Q-026 **Standard and Reagent Management and Traceability** or its equivalent revision or replacement.

6.2. General Analytical Instrument Calibration Procedures (Organic and Inorganic)

6.2.1. All support equipment and instrumentation are calibrated or checked before use to ensure proper functioning and verify that the laboratory's requirements are met. All calibrations are performed by, or under the supervision of, an experienced analyst at scheduled intervals against either certified standards traceable to recognized national standards or reference standards whose values have been statistically validated.

6.2.2. Calibration standards for each parameter are chosen to establish the linear range of the instrument and must bracket the concentrations of those parameters measured in the samples. The lowest calibration standard is the lowest concentration for which quantitative data may be reported. Data reported below this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in a narrative. The highest calibration standard is the highest concentration for which quantitative data may be reported. Data reported above this level is considered to have less certainty and must be reported using appropriate data qualifiers or explained in the narrative. Any specific method requirement for number and type of calibration standards supersedes the general requirement. Instrument and method specific calibration criteria are explained within the specific analytical standard operating procedures for each facility.

6.2.3. Results from all calibration standards analyzed must be included in constructing the calibration curve with the following exceptions:

6.2.3.1. The lowest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done on an individual analyte basis. The reporting limit must be adjusted to the lowest concentration included in the calibration curve;

6.2.3.2. The highest level calibration standard may be removed from the calibration as long as the remaining number of concentration levels meets the minimum established by the method and standard operating procedure. For multi-parameter methods, this may be done an individual analyte basis. The upper limit of quantitation must be adjusted to the highest concentration included in the calibration curve;

6.2.3.3. Multiple points from either the high end or the low end of the calibration curve may be excluded as long as the remaining points are contiguous in nature and the minimum number of levels remains as established by method or standard operating procedure. The reporting limit or quantitation range, whichever is appropriate, must be adjusted accordingly;

6.2.3.4. Results from a concentration level between the lowest and highest calibration levels can only be excluded from an initial calibration curve for a documentable and acceptable cause with approval from the responsible department supervisor and the local SQM/QM or their designee. An acceptable cause is defined as an obvious sample introduction issue that resulted in no response, documentation of an incorrectly prepared standard, or a documented response of a single standard that is greater than 2X the difference from the expected value of that standard. The results for all analytes are to be excluded and the point must be replaced by re-analysis. Re-analysis of this interior standard must occur within the same 12-hour tune time period for GC/MS methodologies and within 8 hours of the initial analysis of that standard for non-GC/MS methodologies. All samples analyzed prior to the re-analyzed calibration curve point must be re-analyzed after the calibration curve is completed and re-processed against the final calibration curve.

6.2.4. Instrumentation or support equipment that cannot be calibrated to specification or is otherwise defective is clearly labeled as out-of-service until it has been repaired and tested to demonstrate it meets the laboratory's specifications. All repair and maintenance activities including service calls are documented in the maintenance log. Equipment sent off-site for calibration testing is packed and transported to prevent breakage and is in accordance with the calibration laboratory's recommendations.

6.2.5. In the event that recalibration of a piece of test equipment indicates the equipment may have been malfunctioning during the course of sample analysis, an investigation is performed. The results of the investigation along with a summary of the information reviewed are documented and maintained by the quality manager. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed. This allows for sufficient investigation and review of documentation to determine the impact on the analytical results. Instrumentation found to be consistently out of calibration is either repaired and positively verified or taken out of service and replaced.

6.2.6. Raw data records are retained to document equipment performance. Sufficient raw data is retained to reconstruct the instrument calibration and explicitly connect the continuing calibration verification to the initial calibration.

6.2.7. **General Organic Calibration Procedures**

6.2.7.1. Calibration standards are prepared at a minimum of five concentrations for organic analyses (unless otherwise stipulated in the method).

6.2.7.2. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Curves that do not meet the appropriate criteria require corrective action that may include re-running the initial calibration curve. Rounding to meet initial calibration criteria is not allowed, that is, 15.3 cannot be rounded down to meet a $\leq$ 15% RSD requirement. This also applies to linear and non-linear fit requirements. All initial calibrations are verified with an initial calibration verification standard (ICV) obtained from a second manufacturer or second lot from the same manufacturer if that lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

6.2.7.3. The calibration curve is periodically verified by the analysis of a mid-level continuing calibration verification (CCV) standard during the course of sample analysis. This standard is from the same source as the initial calibration unless otherwise specified in the source method to be from an alternate source material. Rounding to meet continuing calibration criteria is not allowed. Continuing calibration verification is performed at the beginning and end of each analytical batch except if an internal standard is used, then only one verification at the beginning of the batch is needed, whenever it is expected that the analytical system may be out of calibration, if the time period for calibration has expired, or for analytical systems that have specific calibration verification requirements. This verification standard must meet acceptance criteria in order for sample analysis to proceed.

6.2.7.4. In the event that the CCV does not meet the acceptance criteria, a second CCV may be injected as part of the diagnostic evaluation and corrective action investigation. If the second CCV is acceptable, the analytical sequence may be continued. If both CCVs fail, the analytical sequence is terminated and corrective action is initiated. Sample analysis cannot begin until after documented corrective action has been completed and either two consecutive passing CCVs have been analyzed or the instrument has successfully passed a new initial calibration. All samples analyzed since the last compliant CCV are re-analyzed for methodologies utilizing external calibration.

6.2.7.5. When instruments are operating unattended, autosamplers may be programmed to inject consecutive CCVs as a preventative measure against CCV failure with no corrective action. In this case, both CCVs must be evaluated to determine potential impact to the results. A summary of the decision tree and necessary documentation are listed below:

- If both CCVs meet the acceptance criteria, the analytical sequence is allowed to continue without corrective action. The 12 hour clock begins with the injection of the second CCV.
- If the first CCV does not meet the acceptance criteria and the second CCV is acceptable, the analytical sequence is continued and the results are reported.
- If the first CCV meets the acceptance criteria and the second CCV is out of control, the samples after the out of control CCV must be re-analyzed in a compliant analytical sequence.
- If both CCVs are out of control, all samples since the last acceptable CCV must be re-analyzed in a compliant analytical sequence.

6.2.7.6. Some analytical methods require that samples be bracketed by passing CCVs analyzed both before and after the samples. This is specific to each method but, as a general rule, all external calibration methods require bracketing CCVs. Most internal standard calibrations do not require bracketing CCVs.

6.2.7.7. Some analytical methods require verification based on a time interval; some methods require a frequency based on an injection interval. The type and frequency of the calibration verifications is dependent on both the analytical method and possibly on the quality program associated with the samples. The type and frequency of calibration verification will be documented in the method specific SOP employed by each laboratory.

6.2.8. General Inorganic Calibration Procedures

6.2.8.1. The instrument is initially calibrated with standards at multiple concentrations to establish the linearity of the instrument's response. A calibration blank is also included. Initial calibration curves are evaluated against appropriate statistical models as required by the analytical methods. Rounding to meet initial calibration criteria is not allowed. This also applies to linear and non-linear fit requirements. The number of calibration standards used depends on the specific method criteria or customer project requirements, although normally a minimum of three standards is used.

6.2.8.2. The ICP and ICP/MS can be standardized with a zero point and a single point calibration if:

- Prior to analysis, the zero point and the single point calibration are analyzed and a linear range has been established,
- Zero point and single point calibration standards are analyzed with each batch
- A standard corresponding to the LOQ is analyzed with the batch and meets the established acceptance criteria
- The linearity is verified at the frequency established by the method or manufacturer.

6.2.8.3. All initial calibrations are verified with an initial calibration verification standard (ICV) obtained from a second manufacturer or second lot from the same manufacturer if the lot can be demonstrated as prepared independently from other lots prior to the analysis of samples. Sample results are quantitated from the initial calibration unless otherwise required by regulation, method, or program.

6.2.8.4. During the course of analysis, the calibration curve is periodically verified by the analysis of calibration verification standards (CCV). A calibration verification standard is analyzed within each analytical batch at method/program specific intervals to verify that the initial calibration is still valid. The CCV is also analyzed at the end of the analytical batch.

6.2.8.5. A calibration blank is also run with each calibration verification standard to verify the cleanliness of the system. All reported results must be bracketed by acceptable CCVs. Instrument and method specific calibration acceptance criteria are explained within the specific analytical standard operating procedures for each facility.

6.2.8.6. Interference check standards are also analyzed per method requirements and must meet acceptance criteria for metals analyses.

6.3. Support Equipment Calibration Procedures

6.3.1. All support equipment is calibrated or verified at least annually using NIST traceable references over the entire range of use. The results of calibrations or verifications must be within the specifications required or the equipment will be removed from service until repaired. The laboratory maintains records to demonstrate the correction factors applied to working thermometers.

6.3.2. On each day the equipment is used, balances, ovens, refrigerators (those used to keep samples and standards at required temperatures), freezers, and water baths are checked in the expected use range with NIST traceable references in order to ensure the equipment meets laboratory specifications and these checks are documented appropriately.

6.3.3. **Analytical Balances**

6.3.3.1. Each analytical balance is calibrated or verified at least annually by a qualified service technician. The calibration of each balance is verified each day of use with weights traceable to NIST bracketing the range of use. Calibration weights are ASTM Class 1 or other class weights that have been calibrated against a NIST standard weight and are re-certified every 5 years at a minimum against a NIST traceable reference. Some accrediting agencies may require more frequent checks. If balances are calibrated by an external agency, verification of their weights must be provided. All information pertaining to balance maintenance and calibration is recorded in the individual balance logbook and/or is maintained on file in the Quality department.

6.3.4. **Thermometers**

6.3.4.1. Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are re-certified, at a minimum, every 3 years with equipment directly traceable to NIST.

6.3.4.2. Working thermometers are compared with the reference thermometers annually according to corporate metrology procedures. Each thermometer is individually numbered and assigned a correction factor based on the NIST reference source. In addition, working thermometers are visually inspected by laboratory personnel prior to use and temperatures are documented.

6.3.4.3. Laboratory thermometer inventory and calibration data are maintained in the Quality department.

6.3.5. **pH/Electrometers**

6.3.5.1. The meter is calibrated before use each day, using fresh buffer solutions. Additional information regarding pH/Electrometers can be found in SOP S-GB-I-071 *Measurement of pH in Water, Soil and Waste* or its equivalent revision or replacement.

6.3.6. **Spectrophotometers**

6.3.6.1. During use, spectrophotometer performance is checked at established frequencies in analysis sequences against initial calibration verification (ICV) and continuing calibration verification (CCV) standards.

6.3.7. **Mechanical Volumetric Dispensing Devices**

6.3.7.1. Mechanical volumetric dispensing devices including bottle top dispensers (those that are critical in measuring a required amount of reagent), pipettes, and burettes, excluding Class A volumetric glassware, are checked for accuracy on a quarterly basis.

6.3.7.2. Additional information regarding calibration and maintenance of laboratory support equipment can be found in SOP S-GB-Q-030 **Support Equipment** or its equivalent revision or replacement.

6.4. Instrument/Equipment Maintenance

6.4.1. The objectives of the Pace Analytical maintenance program are twofold: to establish a system of instrument care that maintains instrumentation and equipment at required levels of calibration and sensitivity, and to minimize loss of productivity due to repairs.

6.4.2. The Operations Manager and/or department manager/supervisors are responsible for providing technical leadership to evaluate new equipment, solve equipment problems, and coordinate instrument repair and maintenance. Analysts have the primary responsibility to perform routine maintenance.

6.4.3. To minimize downtime and interruption of analytical work, preventative maintenance is routinely performed on each analytical instrument. Up-to-date instructions on the use and maintenance of equipment are available to staff in the department where the equipment is used.

6.4.4. Department manager/supervisors are responsible for maintaining an adequate inventory of spare parts required to minimize equipment downtime. This inventory includes parts and supplies that are subject to frequent failure, have limited lifetimes, or cannot be obtained in a timely manner should a failure occur.

6.4.5. All major equipment and instrumentation items are uniquely identified to allow for traceability. Equipment/instrumentation is, unless otherwise stated, identified as a system and not as individual pieces. The laboratory maintains equipment records that include the following:

- The name of the equipment and its software
- The manufacturer's name, type, and serial number
- Approximate date received and date placed into service
- Current location in the laboratory
- Condition when received (new, used, etc.)
- Copy of any manufacturer's manuals or instructions
- Dates and results of calibrations and next scheduled calibration (if known)
- Details of past maintenance activities, both routine and non-routine
- Details of any damage, modification or major repairs

6.4.6. All instrument maintenance is documented in maintenance logbooks that are assigned to each particular instrument or system.

6.4.7. The maintenance log entry must include a summary of the results of that analysis and verification by the analyst that the instrument has been returned to an in-control status. In addition, each entry must include the initials of the analyst making the entry, the dates the maintenance actions were performed, and the date the entry was made in the maintenance logbook, if different from the date(s) of the maintenance.

6.4.8. Any equipment that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown to be defective, is taken out of service and clearly identified. The equipment shall not be used to analyze customer samples until it has been repaired and shown to perform satisfactorily. In the event of instrumentation failure, to avoid hold time issues, the lab may subcontract the necessary samples to another Pace lab or to an outside subcontract lab if possible.

7.0. CONTROL OF DATA

Analytical results processing, verification, and reporting are procedures employed that result in the delivery of defensible data. These processes include, but are not limited to, calculation of raw data into final concentration values, review of results for accuracy, evaluation of quality control criteria and assembly of technical reports for delivery to the data user.

All analytical data undergo a well-defined, well-documented multi-tier review process prior to being reported to the customer. This section describes procedures used by PASI for translating raw analytical data into accurate final sample reports as well as PASI data storage policies.

7.1. Analytical Results Processing

7.1.1. When analytical, field, or product testing data is generated, it is either recorded in a bound laboratory logbook (e.g., Run log or Instrument log) or copies of computer-generated printouts that are appropriately labeled and filed. These logbooks and other laboratory records are kept in accordance with each facility's Standard Operating Procedure for documentation storage and archival. If the laboratory chooses to minimize or eliminate its paper usage, these records can be kept on electronic media. In this case, the laboratory must ensure that there are sufficient redundant electronic copies so no data is lost due to unforeseen computer issues.

7.1.2. The primary analyst is responsible for initial data reduction and review. This includes confirming compliance with required methodology, verifying calculations, evaluating quality control data, noting non-conformances in logbooks or as footnotes or narratives, and uploading analytical results into the LIMS. The primary analyst must be clearly identified in all applicable logbooks, spreadsheets and LIMS fields.

7.1.3. The primary analyst then compiles the initial data package for verification. This compilation must include sufficient documentation for data review. It may include standard calibrations, chromatograms, manual integration documentation, electronic printouts, chain of custody forms, and logbook copies.

7.1.4. Some agencies or customers require different levels of data reporting. For these special levels, the primary analyst may need to compile additional project information, such as initial calibration data or extensive spectral data, before the data package proceeds to the verification step.

7.2. Data Verification

7.2.1. Data verification is the process of examining data and accepting or rejecting it based on pre-defined criteria. This review step is designed to ensure that reported data are free from calculation and transcription errors, that quality control parameters are evaluated, and that any non-conformances are properly documented.

7.2.2. Analysts performing the analysis and subsequent data reduction have primary responsibility for quality of the data produced. The primary analyst initiates the data verification process by reviewing and accepting the data, provided QC criteria have been met for the samples being reported. Data review checklists, either hardcopy or electronic, are used to document the data review process. The primary analyst is responsible for the initial input of the data into the LIMS. The primary analyst and reviewer must be clearly identified on all applicable data review checklists.

7.2.3. The completed data package is then sent to a designated qualified reviewer (this cannot be the primary analyst). The following criteria have been established to qualify someone as a data reviewer. To perform secondary data review, the reviewer must:

7.2.3.1. Have a current Demonstration of Capability (DOC) study on file and have an SOP acknowledgement form on file for the method/procedure being reviewed; or, See Note

7.2.3.2. Have a DOC on file for a similar method/technology (i.e., GC/MS) and have an SOP acknowledgment form on file for the method/procedure being reviewed; or, See Note

7.2.3.3. Supervise or manage a Department and have an SOP acknowledgment form on file for the method/procedure being reviewed; or,

7.2.3.4. Have significant background in the department/methods being reviewed through education or experience and have an SOP acknowledgment form on file for the method/procedure being reviewed.

7.2.4. **Note:** Secondary reviewer status must be approved personally by the SQM/QM or SGM/GM/AGM/OM in the event that this person has no prior experience on the specific method or general technology.

7.2.5. This reviewer provides an independent technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. This assessment involves a quality control review for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation. The reviewer validates the data entered into the LIMS and documents approval of manual integrations.

7.2.6. Once the data have been technically reviewed and approved, authorization for release of the data from the analytical section is indicated by initialing and dating the data review checklist or otherwise initialing and dating the data (or designating the review of data electronically). The Operations or Project Manager examines the report for method appropriateness, detection limits and QC acceptability. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution. Alternately, final reports can be set to auto email to the client after the analytical results are final and have been run through the Data Checker program for errors. These are set up on a case-by-case basis.

7.2.7. Additional information regarding data review procedures can be found in SOP S-GB-Q-032 **Data Review** or its equivalent revision or replacement, as well as in SOP S-GB-Q-024 **Manual Integration** or its equivalent revision or replacement.

7.2.8. The Data Checker program will process validated data for a given project against a set of pre-determined requirements and known chemistry relationships. The program creates a report that includes a series of warnings and errors for any requirement or condition determined to be suspect or incorrect. These warnings and error counts are displayed on the “Project Inquiry by Client” screen and on the final Data Checker reports. For projects that have any number of warnings or errors, the Data Checker report will provide a message that identifies the source and condition of the error or warning.

7.2.9. Some reports and/or data packages may be reviewed by the QM or SQM or designee based on program requirements (e.g., DoD) or client requirements. In this case a thorough review for completeness and accuracy may include a compilation of raw data and QC summaries in addition to the final report to produce a full deliverable package. In the case of DoD, 100% of all packages must have a final administrative review (to confirm that primary and secondary reviews were completed and documented and that data packages are complete) and 10% of all data packages must be reviewed by the

Quality Manager for technical completeness/accuracy. This 10% review can be done after the data packages have been submitted to the clients. See SOP SOT-ALL-Q-011 (or equivalent replacement), **Audits and Inspections**, for full Quality department final report and raw data review requirements.

7.3. Data Reporting

7.3.1. Data for each analytical fraction pertaining to a particular PASI project number are delivered to the Project Manager for assembly into the final report. All points mentioned during technical and QC reviews are included in data qualifiers on the final report or in a separate case narrative if there is potential for data to be impacted.

7.3.2. Final reports are prepared according to the level of reporting required by the customer and can be transmitted to the customer via hardcopy or electronic deliverable. A standard PASI final report consists of the following components:

7.3.2.1. A title which designates the report as “Final Report”, “Laboratory Results”, “Certificate of Results”, etc.;

7.3.2.2. Name and address of laboratory (or subcontracted laboratories, if used);

7.3.2.3. Phone number and name of laboratory contact to where questions can be referred;

7.3.2.4. A unique identification number for the report. The pages of the report shall be numbered and a total number of pages shall be indicated;

7.3.2.5. Name and address of customer and name of project;

7.3.2.6. Unique identification of samples analyzed as well as customer sample IDs;

7.3.2.7. Identification of any sample that did not meet acceptable sampling requirements of the relevant governing agency, such as improper sample containers, holding times missed, sample temperature, etc.;

7.3.2.8. Date and time of collection of samples, date of sample receipt by the laboratory, dates of sample preparation and analysis, and times of sample preparation and analysis when the holding time for either is 72 hours or less;

7.3.2.9. Identification of the test methods used;

7.3.2.10. Identification of sampling procedures if sampling was conducted by the laboratory;

7.3.2.11. Deviations from, additions to, or exclusions from the test methods. These can include failed quality control parameters, deviations caused by the matrix of the sample, etc., and can be shown as a case narrative or as defined footnotes to the analytical data;

7.3.2.12. Identification of whether calculations were performed on a dry or wet-weight basis;

7.3.2.13. Reporting limits used;

7.3.2.14. Final results or measurements, supported by appropriate chromatograms, charts, tables, spectra, etc.;

7.3.2.15. A signature and title, electronic or otherwise, of person accepting responsibility for the content of the report;

7.3.2.16. Date report was issued;

7.3.2.17. A statement clarifying that the results of the report relate only to the samples tested or to the samples as they were received by the laboratory;

7.3.2.18. If necessary, a statement indicating that the report must not be reproduced except in full, without the written approval of the laboratory;

7.3.2.19. Identification of all test results provided by a subcontracted laboratory or other outside source;

7.3.2.20. Identification of results obtained outside of quantitation levels.

In addition to the requirements listed above, final reports shall also contain the following items when necessary for the interpretation of results:

7.3.2.21. Deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions;

7.3.2.22. Where relevant, a statement of compliance/non-compliance with requirements and/or specifications (e.g., the TNI standard);

7.3.2.23. Where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer's instruction so requires, or when the uncertainty affects compliance to a specification limit;

7.3.2.24. Where appropriate and needed, opinions and interpretations, which may include opinions on the compliance/non-compliance of the results with requirements, fulfillment of contractual requirements, recommendations on how to use the results, and guidance to be used for improvement;

7.3.3. Additional items may be required per Client QAPPs or different state regulations, i.e. Affidavit for Ohio VAP reports.

7.3.4. Any changes made to a final report shall be designated as "Revised" or equivalent wording. The laboratory must keep sufficient archived records of all laboratory reports and revisions. For higher levels of data deliverables, a copy of all supporting raw data is sent to the customer along with a final report of results. When possible, the PASI facility will provide electronic data deliverables (EDD) as required by contracts or upon customer request.

7.3.5. Customer data that requires transmission by telephone, telex, facsimile or other electronic means undergoes appropriate steps to preserve confidentiality.

7.3.6. The following positions are the only approved signatories for PASI final reports:

- Senior General Manager
- General Manager
- Assistant General Manager
- Senior Quality Manager
- Quality Manager
- Client Services Manager
- Project Manager
- Project Coordinator

7.4. Data Security

7.4.1. All data including electronic files, logbooks, extraction/digestion/distillation worksheets, calculations, project files and reports, and any other information used to produce the technical report are maintained secured and retrievable by the PASI facility.

7.5. Data Archiving

7.5.1. All records compiled by PASI are maintained legible and retrievable and stored secured in a suitable environment to prevent loss, damage, or deterioration by fire, flood, vermin, theft, and/or environmental deterioration. Records are retained for a minimum of five years unless superseded by federal, state, contractual, and/or accreditation requirements. These records may include, but are not limited to, customer data reports, calibration and maintenance of equipment, raw data from instrumentation, quality control documents, observations, calculations, and logbooks. These records are retained in order to provide for possible historical reconstruction including sampling, receipt, preparation, analysis, and personnel involved. TNI-related records will be made readily available to accrediting authorities. Access to archived data is documented and controlled by the SQM/QM or a designated Data Archivist.

7.5.2. Records that are computer generated have either a hard copy or electronic write protected backup copy. Hardware and software necessary for the retrieval of electronic data is maintained with the applicable records. Archived electronic records are stored protected against electronic and/or magnetic sources.

7.5.3. In the event of a change in ownership, accountability or liability, reports of analyses performed pertaining to accreditation will be maintained by the acquiring entity for a minimum of five years. In the event of bankruptcy, laboratory reports and/or records will be transferred to the customer and/or the appropriate regulatory entity upon request.

7.6. Data Disposal

7.6.1. Data that has been archived for the facility's required storage time may be disposed of in a secure manner by shredding, returning to customer, or utilizing some other means that does not jeopardize data confidentiality. Records of data disposal will be archived for a minimum of five years unless superseded by federal, contractual, and/or accreditation requirements. Data disposal includes any preliminary or final reports that are disposed.

8.0. QUALITY SYSTEM AUDITS AND REVIEWS

8.1. Internal Audits

8.1.1. Responsibilities

8.1.1.1. The SQM/QM is responsible for designing and/or conducting internal audits in accordance with a predetermined schedule and procedure. Since internal audits represent an independent assessment of laboratory functions, the auditor must be functionally independent from laboratory operations to ensure objectivity. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation. The SQM/QM evaluates audit observations and verifies the completion of corrective actions. In addition, a periodic corporate audit will be conducted. The corporate audits will focus on the effectiveness of the Quality System as outlined in this manual but may also include other quality programs applicable to an individual laboratory.

8.1.2. Scope and Frequency of Internal Audits

8.1.2.1. The complete internal audit process consists of the following four sections:

- Raw Data Review audits- conducted according to a schedule per local SQM/QM. A certain number of these data review audits are conducted per quarter to accomplish this yearly schedule;
- Quality System audits- considered the traditional internal audit function and includes analyst interviews to help determine whether practice matches method requirements and SOP language;
- Final Report reviews;
- Corrective Action Effectiveness Follow-up.

8.1.2.2. Internal systems audits are conducted yearly at a minimum. The scope of these audits includes evaluation of specific analytical departments or a specific quality related system as applied throughout the laboratory.

8.1.2.3. Where the identification of non-conformities or departures cast doubt on the laboratory's compliance with its own policies and procedures, the lab must ensure that the appropriate areas of activity are audited as soon as possible. Examples of system-wide elements that can be audited include:

- Quality Systems documents, such as Standard Operating Procedures, training documents, Quality Assurance Manual, and all applicable addenda
- Data records and non-technical documents
- Personnel and training files.
- General laboratory safety protocols.
- Chemical handling practices, such as labeling of reagents, solutions, and standards as well as all associated documentation.
- Documentation concerning equipment and instrumentation, calibration/maintenance records, operating manuals.
- Sample receipt and management practices.
- Analytical documentation, including any discrepancies and corrective actions.

- General procedures for data security, review, documentation, reporting, and archiving.
- Data integrity issues such as proper manual integrations.

8.1.2.4. When the operations of a specific department are evaluated, a number of additional functions are reviewed including:

- Detection limit studies
- Internal chain of custody documentation
- Documentation of standard preparations
- Quality Control limits and Control charts

8.1.2.5. Certain projects may require an internal audit to ensure laboratory conformance to site work plans, sampling and analysis plans, QAPPs, etc.

8.1.2.6. A representative number of data audits are completed annually. Findings from these data audits are handled in the same manner as those from other internal and external audits.

8.1.2.7. The laboratory, as part of their overall internal audit program, ensures that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery and reporting of potential data integrity issues are handled in a confidential manner. All investigations that result in findings of inappropriate activity are fully documented, including the source of the problem, the samples and customers affected the impact on the data, the corrective actions taken by the laboratory, and which final reports had to be re-issued. Customers must be notified within 30 days after the data investigation is completed and the impact to final results is assessed.

8.1.3. Internal Audit Reports and Corrective Action Plans

8.1.3.1. Additional information can be found in SOP S-GB-Q-022 **Internal and External Audits** or its equivalent revision or replacement.

8.1.3.2. A full description of the audit, including the identification of the operation audited, the date(s) on which the audit was conducted, the specific systems examined, and the observations noted are summarized in an internal audit report. Although other personnel may assist with the performance of the audit, the SQM/QM writes and issues the internal audit report identifying which audit observations are deficiencies that require corrective action.

8.1.3.3. When audit findings cast doubt on the effectiveness of the operations or on the correctness of validity of the laboratory's environmental test results, the laboratory will take timely corrective action and notify the customer in writing within three business days, if investigations show that the laboratory results may have been affected.

8.1.3.4. Once completed, the internal audit report is issued jointly to the SGM/GM/AGM/OM and the manager(s)/supervisor(s) of the audited operation at a minimum. The responsible manager(s)/supervisor(s) responds within 14 days with a proposed plan to correct all of the deficiencies cited in the audit report. The SQM/QM may grant additional time for responses to large or complex deficiencies (not to exceed 30 days). Each response must include timetables for completion of all proposed corrective actions.

8.1.3.5. The SQM/QM reviews the audit responses. If the response is accepted, the SQM/QM uses the action plan and timetable as a guideline for verifying completion of the corrective action(s). If the SQM/QM determines that the audit response does not adequately address the correction of cited deficiencies, the response will be returned for modification.

8.1.3.6. To complete the audit process, the SQM/QM performs a re-examination of the areas where deficiencies were found to verify that all proposed corrective actions have been implemented. An audit deficiency is considered closed once implementation of the necessary corrective action has been audited and verified. This is usually within 60-90 days after implementation. If corrective action cannot be verified, the associated deficiency remains open until that action is completed.

8.2. External Audits

8.2.1. PASI laboratories are audited regularly by regulatory agencies to maintain laboratory certifications and by customers to maintain appropriate specific protocols.

8.2.2. Audit teams external to the company review the laboratory to assess the effectiveness of systems and degree of technical expertise. The SQM/QM and other QA staff host the audit team and assist in facilitation of the audit process. Generally, the auditors will prepare a formalized audit report listing deficiencies observed and follow-up requirements for the laboratory. In some cases, items of concern are discussed during a debriefing convened at the end of the on-site review process.

8.2.3. The laboratory staff and supervisors develop corrective action plans to address any deficiencies with the guidance of the SQM/QM. The SGM/GM/AGM/OM provides the necessary resources for staff to develop and implement the corrective action plans. The SQM/QM collates this information and provides a written response to the audit team. The response contains the corrective action plan and expected completion dates for each element of the plan. The SQM/QM follows-up with the laboratory staff to ensure corrective actions are implemented and that the corrective action was effective.

8.3. Quarterly Quality Reports

8.3.1. The SQM/QM is responsible for preparing a quarterly report to management summarizing the effectiveness of the laboratory Quality Systems. This status report will include:

- Overview of quality activities for the quarter
- Certification status
- Proficiency Testing study results
- SOP revision activities
- Internal audit (method/system) findings
- Manual integration audit findings (Mintminer)
- Raw Data and Final Report review findings
- MDL activities
- Other significant Quality System items

8.3.2. The Corporate Director of Quality utilizes the information from each laboratory to make decisions impacting the quality program compliance of the company as a whole. Each

SGM/GM/AGM/OM utilizes the quarterly report information to make decisions impacting Quality Systems and operational systems at a local level.

8.3.3. Additional information can be found in SOP S-ALL-Q-014 **Quality System Review** or its equivalent revision or replacement.

8.4. Annual Managerial Review

8.4.1. A managerial review of Management and Quality Systems is performed on an annual basis at a minimum. This allows for assessing program effectiveness and introducing changes and/or improvements.

8.4.2. The managerial review must include the following topics of discussion:

- Suitability of quality management policies and procedures
- Manager/Supervisor reports
- Internal audit results
- Corrective and preventive actions
- External assessment results
- Proficiency testing studies
- Sample capacity and scope of work changes
- Customer feedback, including complaints
- Recommendations for improvement,
- Other relevant factors, such as quality control activities, resources, and staffing.

8.4.3. This managerial review must be documented for future reference by the SQM/QM and copies of the report are distributed to laboratory staff. Results must feed into the laboratory planning system and must include goals, objectives, and action plans for the coming year. The laboratory shall ensure that any actions identified during the review are carried out within an appropriate and agreed upon timescale.

8.5. Customer Service Reviews

8.5.1. As part of the annual managerial review listed previously, the sales staff is responsible for reporting on customer feedback, including complaints. The acquisition of this information is completed by performing surveys.

8.5.2. The sales staff continually receives customer feedback, both positive and negative, and reports this feedback to the laboratory management in order for them to evaluate and improve their management system, testing activities and customer service.

8.5.3. In addition, the labs must be willing to cooperate with customers or their representatives to clarify customer requests and to monitor the laboratory's performance in relation to the work being performed for the customers. This cooperation may include providing the customer reasonable access to relevant areas of the lab for the witnessing of tests being performed; or the preparation of samples or data deliverables to be used for verification purposes.

8.5.4. Customer service is an important aspect to Pace's overall objective of providing a quality product. Good communication should be provided to the customer's throughout projects. The lab should inform the customer of any delay or major deviations in the performance of analytical tests.

9.0. CORRECTIVE ACTION

Additional information can be found in SOP S-GB-Q-027 **Corrective and Preventive Actions** or its equivalent revision or replacement.

During the process of sample handling, preparation, and analysis, or during review of quality control records, or during reviews of non-technical portions of the lab, certain occurrences may warrant the necessity of corrective actions. These occurrences may take the form of analyst errors, deficiencies in quality control, method deviations, or other unusual circumstances. The Quality System of PASI provides systematic procedures for the documentation, monitoring, completion of corrective actions, and follow-up verification of the effectiveness of these corrective actions. This can be done using PASI's LabTrack system that lists among at a minimum, the deficiency by issue number, the deficiency source, responsible party, root cause, resolution, due date, and date resolved.

9.1. Corrective Action Documentation

9.1.1. The following items are examples of sources of laboratory deviations or non-conformances that warrant some form of documented corrective action:

- Internal Laboratory Non-Conformance Trends
- PE/PT Sample Results
- Internal and External Audits
- Data or Records Review (including non-technical records)
- Client Complaints
- Client Inquiries
- Holding Time violations

9.1.2. Documentation of corrective actions may be in the form of a comment or footnote on the final report that explains the deficiency (e.g., matrix spike recoveries outside of acceptance criteria) or it may be a more formal documentation (either paper system or computerized spreadsheet). This depends on the extent of the deficiency, the impact on the data, and the method or customer requirements for documentation.

9.1.3. The person who discovers the deficiency or non-conformance initiates the corrective action documentation on the Non-Conformance Corrective/ Preventive Action report and/or LabTrack. The documentation must include the affected projects and sample numbers, the name of the applicable Project Manager, the customer name, and the sample matrix involved. The person initiating the corrective action documentation must also list the known causes of the deficiency or non-conformance as well as any corrective/preventative actions that they have taken. Preventive actions must be taken in order to prevent or minimize the occurrence of the situation.

9.1.4. In the event that the laboratory is unable to determine the cause, laboratory personnel and management staff will start a root cause analysis by going through an investigative process. During this process, the following general steps must be taken into account: defining the non-conformance, assigning responsibilities, determining if the condition is significant, and investigating the root cause of the nonconformance. General non-conformance investigative techniques follow the path of the sample through the process looking at each individual step in detail. The root cause must be documented within LabTrack or on the Corrective/Preventive Action Report.

9.1.5. After all the documentation is completed, the routing of the Corrective/Preventive Action Report and /or LabTrack will continue from the person initiating the corrective action, to their immediate supervisor or the applicable Project Manager and finally to the SQM/QM, if applicable, who may be responsible for final review and signoff of corrective/preventive actions.

9.1.6. In the event that analytical testing or results do not conform to documented laboratory policies or procedures, customer requirements, or standard specifications, the laboratory shall investigate the significance of the non-conformance and document appropriate corrective actions. The proper level of laboratory management will review any departure from these requirements for technical suitability. These departures are permitted only with the approval of the SGM/GM/AGM/OM or the SQM/QM. Where necessary, Project Management will notify the customer of the situation and will advise of any ramifications to data quality (with the possibility of work being recalled). The procedures for handling non-conforming work are detailed in SOP S-GB-Q-027 **Corrective and Preventive Actions** or its equivalent revision or replacement.

9.2. Corrective Action Completion

9.2.1. Internal Laboratory Non-Conformance Trends

9.2.1.1. There are several types of non-conformance trends that may occur in the laboratory that would require the initiation of a corrective action report. Laboratories may choose to initiate a corrective action for all instances of one or more of these categories if they so choose, however the intent is that each of these would be handled according to its severity; one time instances could be handled with a footnote or qualifier whereas a systemic problem with any of these categories may require an official corrective action process. These categories, as defined in the Corrective Action SOP are as follows:

- Login error
- Preparation Error
- Contamination
- Calibration Failure
- Internal Standard Failure
- LCS Failure
- Laboratory accident
- Spike Failure
- Instrument Failure
- Final Reporting error

9.2.2. PE/PT Sample Results

9.2.2.1. Any PT result assessed as “not acceptable” requires an investigation and applicable corrective actions. The operational staff is made aware of the PT failures and they are responsible for reviewing the applicable raw data and calibrations and list possible causes for error. The SQM/QM reviews their findings and initiates another external PT sample or an internal PT sample to try and correct the previous failure. Replacement PT results must be monitored by the SQM/QM and reported to the applicable regulatory authorities.

9.2.3. **Internal and External Audits**

9.2.3.1. The SQM/QM is responsible for documenting all audit findings and their corrective actions. This documentation must include the initial finding, the persons responsible for the corrective action, the due date for responding to the auditing body, the root cause of the finding, and the corrective actions needed for resolution. The SQM/QM is also responsible for providing any back-up documentation used to demonstrate that a corrective action has been completed.

9.2.4. **Data Review**

9.2.4.1. In the course of performing primary and secondary review of data or in the case of raw data reviews (e.g., by the SQM/QM), errors may be found which require corrective actions. Any finding that affects the quality of the data requires some form of corrective action, which may include revising and re-issuing of final reports.

9.2.5. **Client Complaints**

9.2.5.1. Project Managers are responsible for issuing corrective action forms, when warranted, for customer complaints. As with other corrective actions, the possible causes of the problem are listed and the form is passed to the appropriate analyst or supervisor for investigation. After potential corrective actions have been determined, the Project Manager reviews the corrective action form to ensure all customer needs or concerns are being adequately addressed.

9.2.6. **Client Inquiries**

9.2.6.1. When an error on the customer report is discovered, the Project Manager is responsible for initiating a formal corrective action form that describes the failure (e.g., incorrect analysis reported, reporting units are incorrect, or reporting limits do not meet objectives). The Project Manager is also responsible for revising the final report if necessary and submitting it to the customer.

9.2.7. **Holding Time Violations**

9.2.7.1. In the event that a holding time has been missed, the analyst or supervisor must complete a formal corrective action form. The Project Manager and the SQM/QM must be made aware of all holding time violations.

9.2.7.2. The Project Manager must contact the customer in order that appropriate decisions are made regarding the hold time excursion and the ultimate resolution is then documented and included in the customer project file.

9.3. Preventive Action Documentation

9.3.1. Pace laboratories can take advantage of several available information sources in order to identify needed improvements in all of their systems including technical, managerial, and quality. These sources may include:

- Management Continuous Improvement Plan (CIP) metrics which are used by all production departments within Pace. When groups compare performance across the company, ways to improve systems may be discovered. These improvements can be made within a department or laboratory-wide.
- Annual managerial reviews- part of this TNI-required and NVLAP-required review is to look at all processes and procedures used by the laboratory over the past year and to determine ways to improve these processes in the future.

- Quality systems reviews- any frequent checks of quality systems (monthly logbook reviews, etc.) can uncover issues that can be corrected or adjusted before they become a larger issue.

9.3.2. When improvement opportunities are identified or if preventive action is required, the laboratory can develop, implement, and monitor preventive action plans.

10.0. GLOSSARY

The source of some of the definitions is indicated previous to the actual definition (e.g., TNI, DoD).

Terms and Definitions	
3P Program	The Pace Analytical continuous improvement program that focuses on Process, Productivity, and Performance. Best Practices are identified that can be used by all PASI labs.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.
Accreditation Body	DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies*. The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.
Analyte	DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.

Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.
Batch	TNI- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples. South Carolina- same definition as TNI except 24 hours should be changed to 8 hours.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of custody Form (COC)	TNI- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100

Confirmation	TNI- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a CVS in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	DoD- Action taken to eliminate a detected non-conformity.

Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration at the 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the sample to a more easily measured form.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.

Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard (DoD QSM), ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).

Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP-AES that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
Internal Standards	TNI- A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.

Laboratory	A body that calibrates and/or tests.
Laboratory Control Sample (LCS)	TNI- (however named, such as laboratory fortified blank (LFB), spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
LabTrack	Database used by Pace Analytical to store and track corrective actions and other laboratory issues.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit(s) of Detection (LOD)	TNI- A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	A quantity of bulk material of similar composition processed or manufactured at the same time.

Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.
Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.
Measurement System	TNI and DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation, test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value, within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.
MintMiner	Program used by Pace Analytical to review large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.

Precision	TNI- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing	TNI- A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.
Proficiency Testing Program	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quality System Matrix	TNI and DoD- These matrix definitions are to be used for purposes of batch and quality control requirements: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard. The quantitation range lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.
Reagent Blank (method reagent blank)	A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.
Reporting Limit Verification Standard (or otherwise named)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall".
Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Sample	Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by Pace Analytical sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.

Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain of custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selective Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.
Should	Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio (S/N)	DoD- S/N is a measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.
Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.

Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes or chemicals of primary concern, identified by the customer on a project-specific basis.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD- A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.

Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty Measurement	The parameter associated with the result of a measurement that characterized the dispersion of the values that could be reasonably attributed to the measurand (i.e. the concentration of an analyte).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results, where failed method or contractual requirements are made to appear acceptable.
Unregulated Contaminate Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminates in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. Note: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

11.0. REFERENCES

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11.2. "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.

11.3. "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.

11.4. U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis.

11.5. U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis.

11.6. "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF.

11.7. "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.

11.8. "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.

11.9. "NIOSH Manual of Analytical Methods", Third Edition, 1984, U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health.

11.10. "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (September 1986).

11.11. Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.

11.12. Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.

11.13. Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.

11.14. Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.

11.15. Requirements for Quality Control of Analytical Data for the Environmental Restoration Program, Martin Marietta, ES/ER/TM-16, December, 1992.

11.16. Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, 1988.

11.17. National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards. Most recent version.

11.18. ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories.

11.19. Department of Defense Quality Systems Manual (QSM), version 5.0, March 2013.

11.20. TNI (The NELAC Institute) Standards; most recent version.

11.21. UCMR3 Laboratory Approval Requirements and Information Document, version 2.0, January 2012.

12.0. REVISIONS

The PASI Corporate Quality Office files both a paper copy and electronic version of a Microsoft Word document with tracked changes detailing all revisions made to the previous version of the Quality Assurance Manual. This document is available upon request. All revisions are summarized in the table below.

Document Number	Reason for Change	Date
Quality Assurance Manual 17.0	Section 2.6.5: Updated facility codes. Section 6.2.3.4: Reworded language regarding calibrations. Section 6.3.7.1: Removed last sentence about syringes. Section 6.4.8: Added sentence about instrumentation failure. Section 7.2.6: Added language regarding auto email function. Section 7.5.1.1: Added red letter section for special data retention requirements. Section 9.2.7.2: Removed sentence regarding hold time reporting by QMs. Section 10: Updated DoD definitions per DoD/DOE QSM, revision 5.0. Also added definitions for LC/MS/MS and UCMR. Section 11: Revised DoD reference and added UCMR3 reference. Attachment VIII: added several drinking water methods and added note 4 regarding hexavalent holding time and preservation.	16April2014

ATTACHMENT I- QUALITY CONTROL CALCULATIONS

PERCENT RECOVERY (%REC)

$$\%REC = \frac{(MSConc - SampleConc)}{TrueValue} * 100$$

NOTE: The SampleConc is zero (0) for theLCS and Surrogate Calculations

PERCENT DIFFERENCE (%D)

$$\%D = \frac{MeasuredValue - TrueValue}{TrueValue} * 100$$

where:
TrueValue = Amount spiked (can also be the $\overline{CF}$ or $\overline{RF}$ of the ICAL Standards)
Measured Value = Amount measured (can also be the CF or RF of the CCV)

PERCENT DRIFT

$$\%Drift = \frac{CalculatedConcentration - TheoreticalConcentration}{TheoreticalConcentrtion} * 100$$

RELATIVE PERCENT DIFFERENCE (RPD)

$$RPD = \frac{|(R1 - R2)|}{(R1 + R2)/2} * 100$$

where:
R1 = Result Sample 1
R2 = Result Sample 2

CORRELATION COEFFICIENT (R)

$$CorrCoeff = \frac{\sum_{i=1}^{N} W_i * (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} W_i * (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} W_i * (Y_i - \overline{Y})^2\right)}}$$

With:		
	N	Number of standard samples involved in the calibration
	i	Index for standard samples
	Wi	Weight factor of the standard sample no. i
	Xi	X-value of the standard sample no. i
	X(bar)	Average value of all x-values
	Yi	Y-value of the standard sample no. i
	Y(bar)	Average value of all y-values

ATTACHMENT I- QUALITY CONTROL CALCULATIONS (CONTINUED)

STANDARD DEVIATION (S)

$$S = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{(n-1)}}$$

where:
n = number of data points
X_i = individual data point
$\overline{X}$ = average of all data points

AVERAGE ($\overline{X}$)

$$\overline{X} = \frac{\sum_{n=1}^{i} X_i}{n}$$

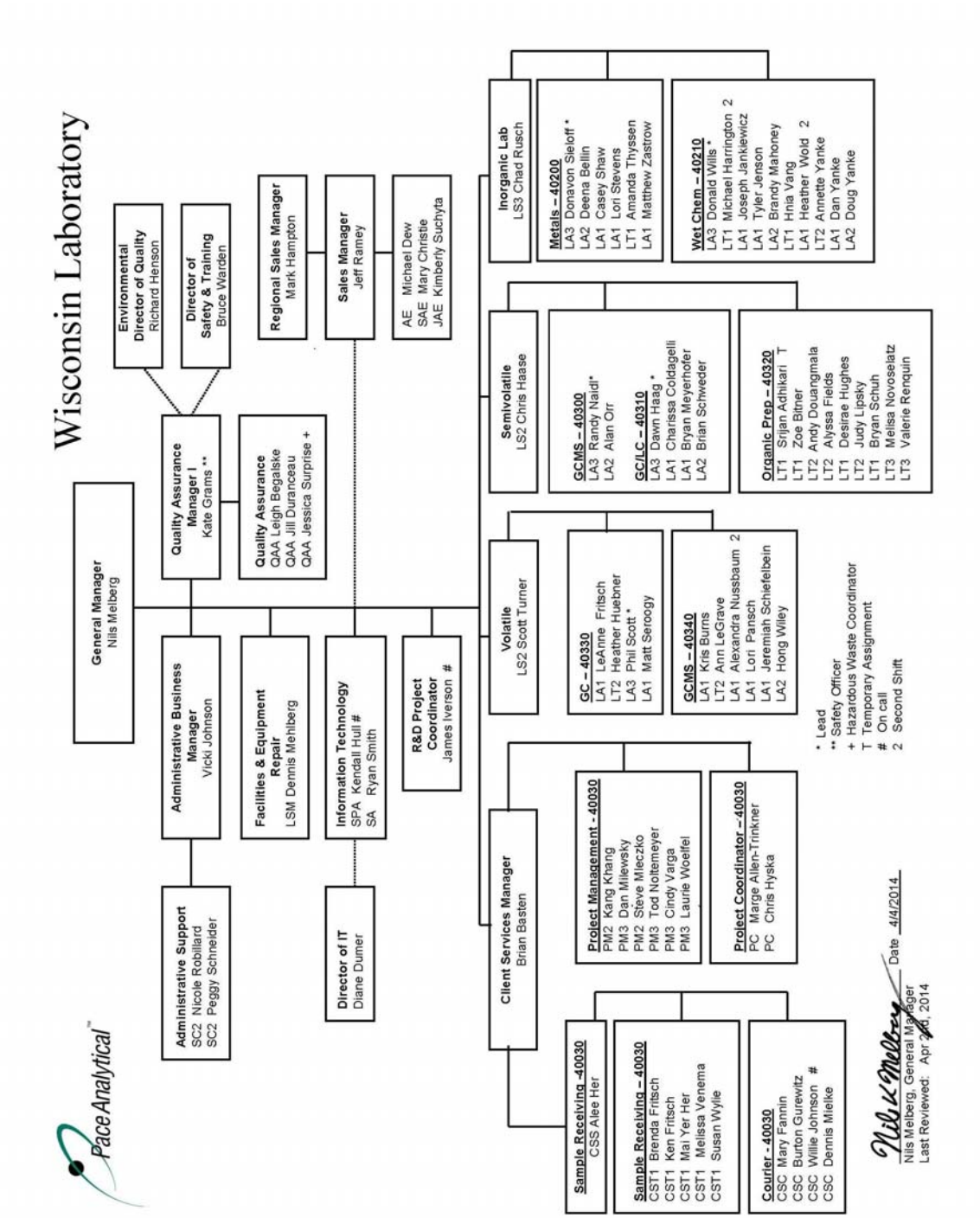
where:
n = number of data points
X_i = individual data point

RELATIVE STANDARD DEVIATION (RSD)

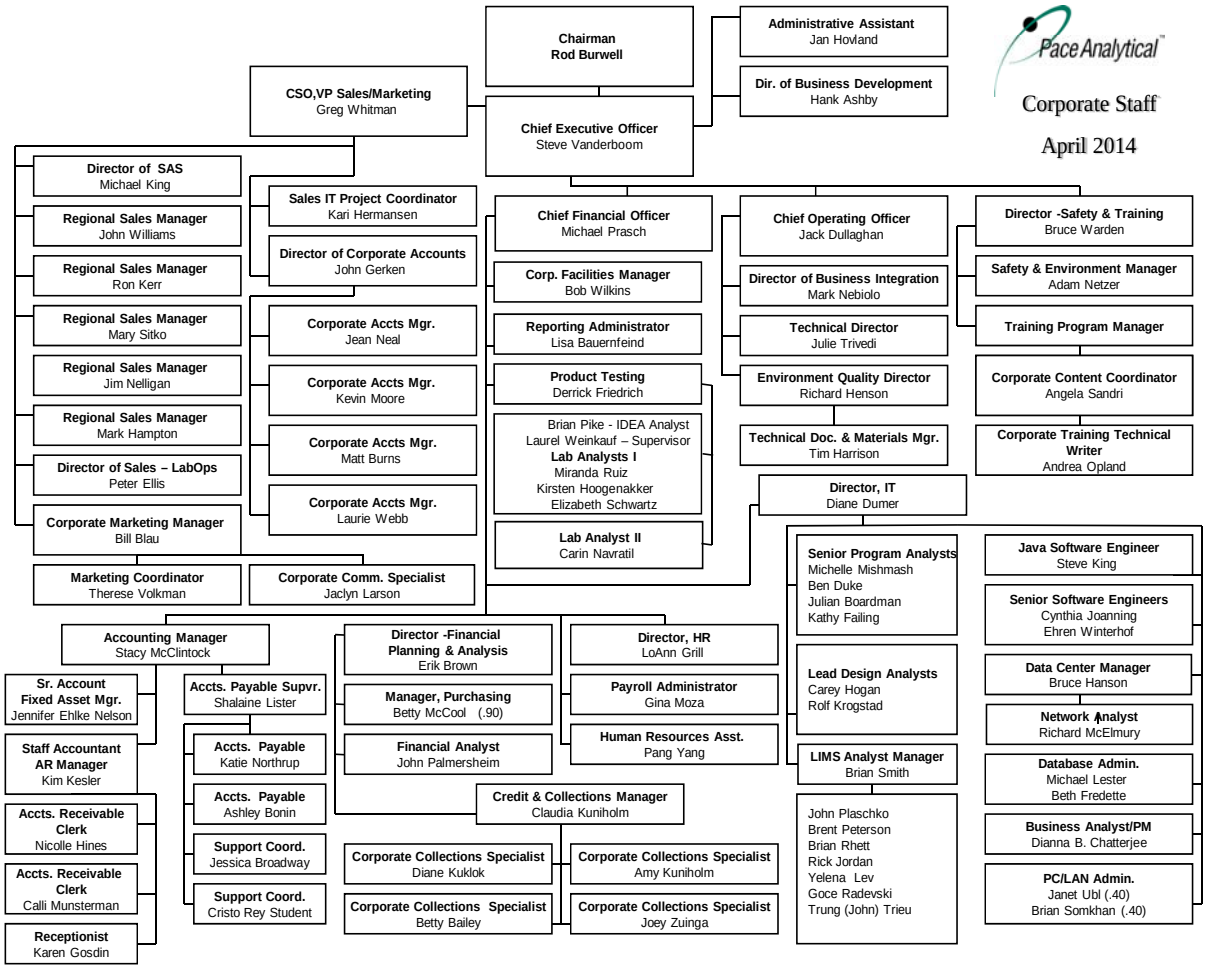
$$RSD = \frac{S}{\overline{X}} * 100$$

where:
S = Standard Deviation of the data points
$\overline{X}$ = average of all data points

ATTACHMENT IIA- LABORATORY ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



ATTACHMENT IIB- CORPORATE ORGANIZATIONAL CHART (CURRENT AS OF ISSUE DATE)



ATTACHMENT III- EQUIPMENT LIST (CURRENT AS OF ISSUE DATE)

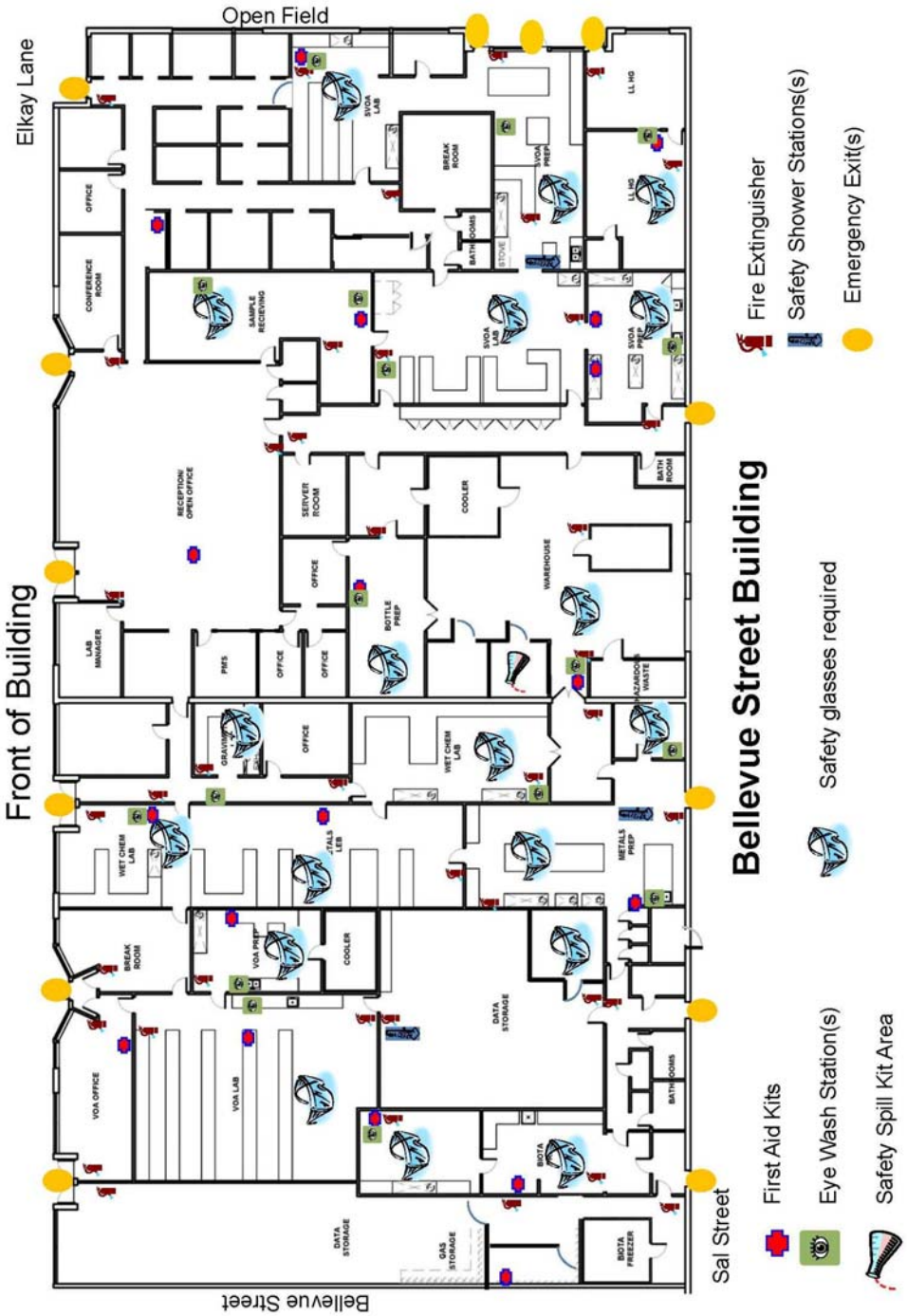
DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
QA	Hot Wire Anemometer	40HWA02	Control Company	4330	-	Air Flow Velocity
SVOA	GC	40GCS1	Hewlett-Packard	5890 Series II	FID	WI-DRO/8015
SVOA	GC	40GCS3	Hewlett-Packard	5890	FID	8015 Alcohol
SVOA	GC	40GCS6	Hewlett-Packard	5890	ECD/ECD	8081
SVOA	GC	40GCS7	Agilent	6890N	ECD/ECD	
SVOA	GC	40GCS8	Hewlett-Packard	6890	ECD/ECD	8081
SVOA	GC	40GCS9	Hewlett-Packard	6890	ECD/ECD	8082
SVOA	GC	40GCSA	Hewlett-Packard	5890	ECD/ECD	Screener
SVOA	GC	40GCSB	Agilent	6890	ECD/ECD	8082
SVOA	GC	40GCSC	Hewlett-Packard	6890	ECD/ECD	8082
SVOA	GC	40GCSE	Hewlett-Packard	5890	ECD/ECD	8082
SVOA	GC	40GCSF	Agilent	7890	FID	WI-DRO/8015
SVOA	GC/MS	40MSS1	Hewlett-Packard	5890	HP 5972	8270C/625
SVOA	GC/MS	40MSS2	Hewlett-Packard	6890	HP 5973	8270C-SIM
SVOA	GC/MS	40MSS3	Hewlett-Packard	6890	HP 5973	8270C-SIM
SVOA	GC/MS	40MSS4	Hewlett-Packard	6890	HP 5973	8270C-SIM
SVOA	GC/MS	40MSS5	Hewlett-Packard	5890	HP 5972	8270 SCREEN
SVOA	GC/MS	40MSS6	Hewlett-Packard	5890	HP 5972	8270C
SVOA	GC/MS	40MSS7	Agilent	7890A	HP 5975	8270C
SVOA	GC/MS	40MSS8	Agilent	7890A	HP 5975C	8270C
SVOA	Separatory Funnel Extractor	40SFE1	Lab Line	-	-	SW846 3510C
SVOA	Separatory Funnel Extractor	40SFE2	Lab Line	-	-	SW846 3510C
SVOA	Separatory Funnel Extractor	40SFE3	Lab Line	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC1	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC2	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC3	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC4	Zymark	-	-	SW846 3510C
SVOA	TurboVap II Concentration Workstation	40TVC5	Zymark	-	-	SW846 3510C
SVOA	Sonicator	40SON3	Lab Line	9334	-	
SVOA	Precision Water Bath	40WB01	Precision	-	-	
SVOA	Water Baths	40WB02	-	-	-	
SVOA	Tube Heaters	-	Kontes	-	-	
SVOA	Accuprep MPS GPC Cleanup System (John)	40GPC2	J2 Scientific	-	-	SW846 3640A
SVOA	Accuprep MPS GPC Cleanup System (Jane)	40GPC1	J2 Scientific	-	-	SW846 3640A
SVOA	Mars Xtraction Microwave System	40MIC1	CEM	907501	-	SW846 3546
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX3	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX4	Gerhardt Soxtherm	SE-30	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX5	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX6	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX7	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX8	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOX9	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Soxtherm Accelerated Soxhlet Extractor	40SOXA	Gerhardt Soxtherm	SE-416	-	SW846 3540C
SVOA	Sonifier Cell Disruptors with Horns	Sonicator #2	-	-	-	SW846 3550B
SVOA	Sonifier Cell Disruptors with Horns	40SON1	Misonix	3000	-	SW846 3550B
SVOA	Sonifier Cell Disruptors with Horns	40SON2	Misonix	S-4000	-	SW846 3550B
SVOA	Environ Shaker	40SKR1	LabLine	3527	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT1	-	-	-	-

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
SVOA	Soxhlet heater mantles and Glassware	40SXT2	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT3	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT4	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT5	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT6	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT7	-	-	-	-
SVOA	Soxhlet heater mantles and Glassware	40SXT8	-	-	-	-
SVOA	Extraction Mixer	40EXT1	-	-	-	-
SVOA	Extraction Mixer	40EXT2	-	-	-	-
SVOA	Extraction Mixer	40EXT3	-	-	-	-
SVOA	Extraction Mixer	40EXT4	-	-	-	-
SVOA	Extraction Mixer	40EXT5	-	-	-	-
VOA	GC/MS	40MSV1	Hewlett-Packard	5890	HP 5972	SW846 8260B/5030
VOA	GC/MS	40MSV2	Hewlett-Packard	6890	HP 5973	SW846 8260B/624/5030B
VOA	GC/MS	40MSV3	Agilent	6850	Agilent 5975	SW846 8260B/624/5030B
VOA	GC/MS	40MSV5	Hewlett-Packard	6890	HP 5973	SW846 8260B/624/5030B
VOA	GC/MS	40MSV7	Hewlett-Packard	6890	HP 5973	SW846 8260B/624/5030B
VOA	GC/MS	40MSV8	Agilent	6850	Agilent 5975B	SW846 8260B/624/5030B
VOA	GC/MS	40MSVA	Hewlett-Packard	7890	Agilent 5975C	SW846 8260B/624/5030B
VOA	GC/MS	40MSVB	Hewlett-Packard	7890A	Agilent 5975C	SW846 8260B/624/5030B
VOA	GC/MS	40MSVC	Hewlett-Packard	7890B	Agilent 5977A	SW846 8260B/624/5030B
VOA	GC	40GCV1	Hewlett-Packard	5890	Agilent 5975	SW846 8021/WI MOD GRO
VOA	GC	40GCV2	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV3	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV4	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV5	Hewlett-Packard	5890	PID/FID	SW846 8021/WI MOD GRO
VOA	GC	40GCV8	Hewlett-Packard	5890	FID	SW846 8015 - MEE
VOA	GC	40SCREEN1	Hewlett-Packard	5890	FID	Screener
VOA	GC	40SCREEN2	Hewlett-Packard	5890	FID	Screener
VOA	SNOOP	40SCREEN3	Research Engineers	-	-	Screener
VOA	Sonicator	40SON4	LabLine	9314	-	VOA Prep
VOA	Sonicator	40SON5	LabLine	9333	-	VOA Prep
WET	Apollo	40WTA5	Tekmar/Dohrmann	Apollo 9000	-	EPA 9060
WET	Fusion	40WTA7	Teledyne	14-9600-100	-	EPA 9060, SM 5310C
WET	Analytik Jena	40WTAA	Analytik Jenna	Multi EA 4000	-	EPA 9060, SM 5310C
WET	SmartChem	40WTA9	Westco Scientific	Smartchem 200		

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
WET	Oxygen Meter	40WET1	YSI	5000	YSI 5010	SM 5210B--01, SM 4500-O G
WET	BOD AUTOEZ	40WETE	Thermo/Orion	10060020	-	SM 5210B
WET	Flashpoint Instrument A	40WET4	Precision	-	-	EPA 1010A
WET	Flashpoint Instrument B	40WET5	Fisher	-	-	EPA 1010A
WET	Turbidimeter	40WET6	Hach	2100P	-	EPA 180.1, SM2130B
WET	Conductivity Meter	40WET7	Accument	30	-	EPA 120.1
WET	pH Meter	40WET8	Orion	720A	-	pH Meter
WET	EH Meter	40WET9	Accument	AB15	-	EH Meter
WET	Radiometer	40WET0	Titration	TIM840		Alkalinity - 2320B
WET	pH meter	40WETB	Symphony	SB20	-	1311, 1312, ASTM D3987
WET	pH Meter	40WETC	Orion	720A	-	pH Meter
WET	pH Meter	40WETD	Corning	320	-	pH Meter
WET	MICRO DIST Rapid Distillation System	40MD1	Lachat	-	-	350.1, 9012A
WET	MICRO DIST Rapid Distillation System	40MD2	Lachat	-	-	350.1, 9012A
WET	MICRO DIST Rapid Distillation System	40MD3	Lachat	-	-	350.1, 9012A
WET	Block Digestion System	40HB01	Westco Scientific	40/20	-	365.4, 351.2
WET	Block Digestion System	40HB02	Tecator	2040	-	365.4, 351.2
WET	Hot Block Digestion System	40HB03	Environmental Express	SC100	-	6010/6020
WET	COD Reactor	40COD1	Hach	45600	-	410.4
WET	COD Reactor	40COD2	Bioscience INC	100 003	-	410.4
WET	Direct Reading Spectrophotometer	40WTA1	Hach	DR 2000	-	EPA 410.4, SM3500Cr-B, Hach 8146, Walkley Black TOC
WET	Quik Chem 8000	40WTA2	Lachat	8000	-	350.1, 365.4, 9012A, 310.2, 351.2, 353.2
WET	Ion Chromatograph	40WTA3	Dionex	DX-120	-	EPA 300.0, 9056A
WET	Ion Chromatograph	40WTAB	Thermo Scientific	ICS-110	-	EPA 300.0, 9056A
WET	Ion Chromatograph	40WTAD	Thermo Scientific (Dionex)	ICS-1100	-	EPA 300.0, 9056A
WET	Quik Chem 8500 Series 2	40WTAC	Lachat	8500 Series 2	-	EPA 353.2, 350.1
WET	Hellige Colony Counter	-	Hellige	-	-	
WET	Steril-Quick Autoclave	-	National	704-9000-D	-	
WET	Stereomaster Stereoscope	-	Fisher	FW 99-20-1385	-	
WET	Sonicator	40SON6	Branson	8210	-	
WET	TCLP Tumbler	40TBL1	Environmental Express	-	-	1311, 1312, ASTM D3987
WET	TCLP Tumbler	40TBL2	Environmental Express	-	-	1311, 1312, ASTM D3987
WET	Reaction Vessel Tumbler	40TBL3	Reaction Vessel	-	-	1311, 1312, ASTM D3987
WET	Centrefudge	40CENT1	International Equipment Co	H260	-	
WET	Buret	40BUR1	KIMAX	253	-	
WET	Buret	40BUR2	Pyrex	2103	-	
MET	Hot Water Bath	40WB05	Thermo Fisher Scientific	2841	-	
MET	ICP	40ICP2	Thermo	ICAP 6500	-	6010
MET	Chiller	40ICP2	Neslab/Thermo	Selex 900	-	6010
MET	ICPMS	40ICM2	Thermo	X Series 2	-	6020
MET	Chiller	40ICM2	Neslab/Thermo	Merlin M75	-	6020
MET	UPS	40ICM2	Toshiba	1600EP		6020
MET	Vacuum Pump	40ICM2	Edwards	EM28		6020
MET	ICPMS	40ICM3	Thermo	X Series 2	-	6020
MET	Chiller	40ICM3	Neslab/Thermo	2500	-	6020
MET	UPS	40ICM3	Toshiba	1600EP		6020
MET	Vacuum Pump	40ICM3	Edwards	EM28		6020
MET	Quick Trace Mercury Analyzer	40HG2	Cetac	M-7500		7470/7471
MET	DMA-80 Direct Mercury Analyzer	40HG4	Milestone	DMA-80	-	7473

DEPT	INSTRUMENT	INSTRUMENT NAME	MANUFACTURER	MODEL NUMBER	DETECTOR	SERVICE ANALYSIS
MET	Hot Block Metals Digestion System	40HB04	Environmental Express	SC100	-	6010/6020
MET	Hot Block Metals Digestion System	40HB06	Environmental Express	SC154	-	6010/6020
MET	Hot Block Metals Digestion System	40HB07	Environmental Express	SC100	-	6010/6020
MET	Hot Block Metals Digestion System	40HB08	Environmental Express	SC196	-	6010/6020
MET	Low-level Mercury Analyzer	40LHG1	Tekran	2500	2500	1631E
MET	Low Level Mercury Analyzer	40LHG4	Analytik Jena	Mercur	-	1631E
MET	Low Level Mercury Analyzer	40LHG5	Cetac	M-8000	-	1631E

ATTACHMENT IV- LABORATORY FLOOR PLAN (CURRENT AS OF ISSUE DATE)



ATTACHMENT V- LABORATORY SOP LIST (CURRENT AS OF ISSUE DATE)

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
MANUALS					
Quality Manual	16.0	Quality Manual	1-Oct-04	6-May-13	Quality
CHP/Safety	9.0	Chemical Hygiene Plan/Safety Manual	18-Jul-11	23-Apr-13	Safety
SAMPLE MAN					
S-ALL-C-002	04	Bottle Order Database	4-Oct-06	4-Jun-13	Sample Management
S-ALL-C-005	03	Operation of PacePort Customer Feedback Form	27-Nov-07	16-Nov-12	Sample Management
S-GB-C-001	04	Procedure to Preserve Samples for Volatile Organic Analysis of Solid Matrices by Method 5035	12-Apr-05	5-Oct-12	Sample Management
S-GB-C-002	04	Extruding Sample from 25 g EnCores for Volatile and Semivolatile Analyses	12-Apr-05	5-Oct-12	Sample Management
S-GB-C-005	05	Maintenance of Ice Chests and Shipping Containers	1-Jun-05	27-Feb-14	Sample Management
S-GB-C-007	04	Laboratory Tracking of Samples	6-Jun-05	4-Oct-13	Sample Management
S-GB-C-008	03	Measurement of Percent Moisture in Soils and Solids	19-Dec-06	5-Oct-12	Sample Management
S-GB-C-009	04	Subcontracting Samples	31-Mar-05	4-Oct-13	Sample Management
S-GB-C-010	05	Sample Management	12-Jul-04	13-May-13	Sample Management
S-GB-C-011	02	Bottle Preparation	4-Oct-06	13-May-13	Sample Management
S-GB-C-012	01	Review of Analytical Requests	23-Dec-08	26-Sep-12	Sample Management
METALS					
S-GB-M-004	04	The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy (7470A/7471A_B)	28-Aug-06	19-Dec-12	Metals
S-GB-M-005	05	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010C	23-Sep-08	19-Dec-12	Metals
S-GB-M-006	04	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020A	25-Sep-08	5-Dec-11	Metals
S-GB-M-007	02	Determination of Trace Metals in Waters and Wastes by Inductively Coupled Plasma Mass Spectroscopy - 6020	13-May-10	5-Dec-11	Metals
S-GB-M-008	02	Cleaning Metals Glassware	5-May-05	21-Sep-12	Metals
S-GB-M-009	02	Hardness by Calculation	27-Oct-05	28-Sep-11	Metals
S-GB-M-013	01	Soil Fraction Preparation for Lead Analysis	21-Mar-06	6-Jul-11	Metals
S-GB-M-014	03	Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010B	28-Feb-07	19-Dec-12	Metals
S-GB-M-015	03	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry (1631E)- Analytik Jena	16-Aug-11	26-Aug-13	Metals
S-GB-M-016	00	Determination of Mercury in Solids by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry	21-Mar-12	21-Mar-12	Metals
S-GB-M-017	01	The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy - CETAC M-7500 (7470A/7471A_B)	3-Aug-11	9-Apr-13	Metals
S-GB-M-018	00	The Determination of Mercury in Biological Samples by Cold Vapor Atomic Absorption Spectroscopy - CETAC M-7500 (245.6)	9-Apr-13	9-Apr-13	Metals
S-GB-M-019	00	Mercury Analysis by Cold-Vapor Atomic Fluorescence Spectrometry CETAC M-8000 (1631E)	26-Aug-13	26-Aug-13	Metals
MICRO					
S-GB-MB-001	01	Fecal Coliform Determination Using the Membrane Filter Technique	13-Sep-05	3-Apr-14	Wetchem
S-GB-MB-002	00	Heterotropic Plate Count	17-Nov-05	5-Oct-12	Wetchem
S-GB-MB-003	00	Colisure Presence/Absence Test for Detection and Identification of Coliform Bacteria and *Escherichia coli* in Drinking Waters	28-Nov-05	5-Oct-12	Wetchem
WET CHEM					
S-GB-I-001	09	Total Sulfide, Iodometric Titration	26-Jan-05	22-Jan-14	Wetchem
S-GB-I-002	07	Flash Point (Pensky-Martens Closed Cup Method For Ignitability	27-Jan-05	28-Jun-13	Wetchem
S-GB-I-009	06	Ion Chromatography- Sulfate, Nitrate, Flouride (EPA 300.0)	7-Feb-05	20-Jan-14	Wetchem
S-GB-I-010	03	Wet Chemistry Glassware Cleaning	5-Feb-05	21-Sep-12	Wetchem
S-GB-I-013	03	Free Liquids (Paint Filter)	13-Jul-05	21-Sep-12	Wetchem
S-GB-I-015	04	Oxidation - Reduction Potential (Eh) Measurement	29-Jul-05	22-Jan-14	Wetchem
S-GB-I-016	04	Specific Gravity	25-Aug-05	22-Jan-14	Wetchem
S-GB-I-017	03	Ferrous Iron	30-Aug-05	28-Sep-12	Wetchem
S-GB-I-020	04	Color Determination in Aqueous Samples	15-Sep-05	28-Sep-12	Wetchem
S-GB-I-027	04	Dissolved Oxygen	1-Mar-06	27-Feb-14	Wetchem
S-GB-I-030	03	Turbidity (Nephelometric)	10-Apr-06	3-Dec-12	Wetchem
S-GB-I-037	04	The Determination of Total Organic Carbon Using the Walkley-Black Procedure	8-Aug-05	21-Sep-12	Wetchem
S-GB-I-044	04	Biochemical Oxygen Demand	15-Feb-07	3-Apr-14	Wetchem
S-GB-I-045	06	Chromium, Hexavalent-Colorimetric	15-Feb-07	22-Jan-14	Wetchem
S-GB-I-047	05	Total Kjeldahl Nitrogen using Block Digestion and Analyzed by Lachat 8000 Flow Injection following EPA Method 351.2	17-Feb-07	20-Jan-14	Wetchem
S-GB-I-048	04	Ammonia using Micro-Distillation and Analyzed by Lachat 8000 Flow Injection following EPA Method 350.1	17-Feb-07	3-Apr-14	Wetchem
S-GB-I-051	04	Nitrate and Nitrite Analyzed by Lachat 8000 Flow Injection	20-Feb-07	20-Jan-14	Wetchem
S-GB-I-052	05	Chemical Oxygen Demand, Colorimetric, Manual (Chemetric Vials)	21-Feb-07	20-Jan-14	Wetchem
S-GB-I-053	02	Acid Volatile Sulfide / Simultaneously Extracted Metals	4-Apr-07	4-Jun-13	Wetchem
S-GB-I-059	04	The Determination of Total Organic Carbon Using the Apollo 9000 Instrument (EPA 9060/A)	21-May-09	28-Sep-12	Wetchem
S-GB-I-061	03	Total Phosphorus using Block Digestion and Analyzed by SmartChem	1-Jun-09	20-Jan-14	Wetchem
S-GB-I-063	04	The Determination of Total Organic Carbon Using the Teledyne Tekmar Fusion Instrument	28-Dec-09	27-Feb-14	Wetchem
S-GB-I-064	02	Cyanide by SmartChem	18-Jan-10	20-Jan-14	Wetchem
S-GB-I-066	01	Measurement of Specific Conductance in Water	12-Dec-06	7-Dec-12	Wetchem

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-I-067	02	TCLP - Toxicity Characteristic Leaching Procedure, SPLP - Synthetic Precipitation Leaching Procedure and ASTM - ASTM D 3987-85	5-Jul-10	13-May-13	Wetchem
S-GB-I-068	02	Measurement of Volatile Solids and Solids in Water	24-Aug-07	24-Dec-13	Wetchem
S-GB-I-069	02	Alkalinity by Titration using the Radiometer TIM840 - SM 2320B	17-Dec-10	19-Sep-13	Wetchem
S-GB-I-070	01	Alkalinity by SmartChem (EPA 310.2)	19-Apr-11	24-Sep-12	Wetchem
S-GB-I-071	01	Measurement of pH in Water, Soil, and Waste	12-Dec-06	28-Sep-12	Wetchem
S-GB-I-072	02	Acidity by Titration using the Radiometer TIM840 - SM 2310B_305.1	1-May-12	19-Sep-13	Wetchem
S-GB-I-073	00	The Determination of Total Organic Carbon Using the EA4000 Instrument (EPA 9060/A)	20-Sep-12	28-Sep-12	Wetchem
S-GB-I-074	01	Biochemical Oxygen Demand by Auto EZ Instrument	20-Jan-14	3-Apr-14	Wetchem
S-GB-I-076	00	The Determination of Total Organic Carbon Using the EA4000 Instrument (Lloyd Kahn)	18-Mar-14	18-Mar-14	Wetchem
GAS SECTION					
S-GB-O-005	04	Soil/Semisolid Sample Preparation for the Analysis of Gasoline Range Organics and Petroleum Volatile Organics by Wisconsin Modified GRO	29-Apr-05	16-Nov-12	Gas
S-GB-O-006	04	Modified Method for Determination of Gasoline Range Organics	27-Apr-05	19-Nov-12	Gas
S-GB-O-008	05	Total Petroleum Hydrocarbons - Gasoline by Gas Chromatography Using Flame-Ionization Detectors	15-Jun-05	10-Dec-12	Gas
S-GB-O-009	04	Aromatic Volatiles by Gas Chromatography Using Photo-Ionization Detectors	27-Apr-05	3-Dec-12	Gas
S-GB-O-010	04	Aqueous Sample Preparation for the Analysis of Gas Range Organics and Petroleum Volatile Organics	29-Apr-05	30-Oct-12	Gas
S-GB-O-017	04	Analysis of Dissolved Methane, Ethane, and Ethene in Ground Water by Static Headspace and Gas Chromatography	27-Apr-05	3-Dec-12	Gas
S-GB-O-063	00	Determination of Gasoline Range Organics by Method AK101	26-Feb-13	6-May-13	Gas
SVOA SECTION					
S-ALL-O-038	01	Processing Tentatively Identified Compounds (TICs) for GC/MS	9-Jun-11	3-Dec-12	QA
S-GB-O-015	05	Cleaning of Glassware Used in the Analysis of Semivolatile Range Organics	11-May-05	27-Jun-13	SVOA
S-GB-O-018	03	Determination of DRO Sample Weight and Methylene Chloride Addition	13-Apr-05	21-Sep-12	SVOA
S-GB-O-019	06	WI Modified Method for Determination of Diesel Range Organics	13-Apr-05	20-Jan-14	SVOA
S-GB-O-023	06	Total Petroleum Hydrocarbons - Diesel	25-May-05	9-Jan-14	SVOA
S-GB-O-025	03	Alcohols & Glycols by Direct Injection GC/FID	14-Dec-05	1-May-12	SVOA
S-GB-O-026	07	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography by 8082	23-May-06	20-Jan-14	SVOA
S-GB-O-027	06	Analysis of Organochlorine Pesticides by Gas Chromatography by 8081	23-May-06	20-Jan-14	SVOA
S-GB-O-028	04	Preparation of Anhydrous Sodium Sulfate and Sand for Extraction Purposes	24-May-06	21-Sep-12	SVOA
S-GB-O-031	03	Extraction of Biological Samples for Organochlorine Pesticides/PCBs	20-Jan-05	21-Sep-12	SVOA
S-GB-O-032	03	Gel Permeation Chromatography	26-Jan-05	21-Sep-12	SVOA
S-GB-O-033	02	Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis	8-Feb-05	21-Sep-12	SVOA
S-GB-O-034	04	Sulfuric Acid Cleanup	3-Feb-05	27-Jun-13	SVOA
S-GB-O-036	04	Florisil Cleanup for PCB and Toxaphene Samples	26-Jan-05	20-Jan-14	SVOA
S-GB-O-037	05	Florisil Cartridge Cleanup for Organochlorine Pesticide Samples	26-Jan-05	4-Apr-14	SVOA
S-GB-O-038	04	Silica Gel Cleanup for Organic Analysis	3-Feb-05	21-Sep-12	SVOA
S-GB-O-039	04	Copper Cleanup for the Removal of Sulfur from PCB and Toxaphene Samples	23-Aug-05	27-Jun-13	SVOA
S-GB-O-040	04	Extraction of Wipes and Oil for PCB Analysis	11-Jul-08	1-May-12	SVOA
S-GB-O-041	05	Extraction of PCBs Using the Automated Soxhlet 3541	23-Aug-07	9-Jan-14	SVOA
S-GB-O-043	03	Extraction of Toxaphene Using the Automated Soxhlet 3541	3-May-07	21-Sep-12	SVOA
S-GB-O-044	02	Determination of Low Level PAHs by GC/MS-SIM in Solid and Biological Matrices:8270SIMPAH	5-Feb-05	13-Jul-11	SVOA
S-GB-O-045	06	Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons, Base/Neutral/Acids, and Total Petroleum Hydrocarbons in Solid Matrices by 3546	21-Sep-07	21-Oct-13	SVOA
S-GB-O-047	03	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography following 8082A	22-Sep-08	21-Sep-12	SVOA
S-GB-O-048	03	Analysis of Fox River Polychlorinated Biphenyls (PCBs) by Gas Chromatography	4-Jun-09	29-Aug-13	SVOA
S-GB-O-049	04	Determination of Semi-Volatile Organics by GC/MS (8270)	28-Nov-06	27-Jun-13	SVOA
S-GB-O-050	03	Determination of Semi-Volatile Organics by GC/MS (Selective Ion Monitoring - 8270C SIM)	28-Nov-06	27-Jan-14	SVOA
S-GB-O-052	01	Extraction of PCBs In Tissue Using the Automated Soxhlet	11-Nov-09	1-May-12	SVOA
S-GB-O-053	02	Separatory Funnel Extraction by 3510C	16-Nov-06	3-May-12	SVOA
S-GB-O-054	01	Ultrasonic Extraction by 3550B	16-Nov-06	1-May-12	SVOA
S-GB-O-055	01	WI Modified Method for Determination of Extended Range Diesel Range Organics	12-Jul-11	1-May-12	SVOA
S-GB-O-057	01	Analysis of Toxaphene by Gas Chromotography Using St.John's River Waste Management Department (SJRWMD) Protocol	10-Jul-10	26-Sep-12	SVOA
S-GB-O-058	01	Analysis of Toxaphene by Gas Chromotography Using Hercules 97 and Total Area Under the Curve (TAUC)	7-Jul-10	26-Sep-12	SVOA
S-GB-O-060	01	Extraction of TPH-Diesel Using the Automated Soxhlet_3541	7-Jul-11	4-Oct-13	SVOA
S-GB-O-061	00	Analysis ofTetrachlorobiphenyls (TCBs) by Gas Chromatography/Mass Spectroscopy	19-Feb-13	19-Feb-13	SVOA
S-GB-O-062	00	SVOA Sample Screening	6-Feb-13	6-Feb-13	SVOA
S-GB-O-064	00	AK102_AK103	27-Mar-13	13-May-13	SVOA
S-GB-O-065	00	Determination of Semi-Volatile Organics by GC/MS (8270D)	5-Dec-13		SVOA
S-GB-O-066	00	Determination of Semi-Volatile Organics by GC/MS (Selective Ion Monitoring - 8270D SIM)	27-Jan-14		SVOA
VOA SECTION					
S-GB-O-001	07	Sample Screening Volatile Organics Prior to Preparation	24-Aug-05	29-Aug-13	VOA
S-GB-O-012	04	Cleaning of Syringes Used in the Analysis of Volatile Organics	12-Apr-05	17-Oct-12	VOA
S-GB-O-056	07	Determination of Volatile Organics by GC/MS 8260	14-Jul-06	27-Jun-13	VOA

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
IT					
S-GB-IT-001	00	System Security and Integrity	26-Jan-05	28-Sep-11	IT
S-GB-IT-002	00	Server Back-up	4-Jun-09	30-Oct-12	IT
QA SYSTEM					
S-ALL-Q-003	08	Document Numbering	12-Apr-04	19-Feb-13	QA
S-ALL-Q-009	06	Laboratory Documentation	22-Dec-04	13-Mar-14	QA
S-ALL-Q-014	04	Quarterly Quality Report	5-May-04	9-Jan-14	QA
S-ALL-Q-015	01	Review of Laboratory Management System	12-Sep-12	4-Jun-13	QA
S-ALL-Q-020	06	Orientation and Training Procedures	30-Jun-05	13-Mar-14	QA
S-ALL-Q-022	03	3P Program Continuous Process Improvement	19-May-05	12-Sep-12	QA
S-ALL-Q-028	03	Use and Operation of Lab Track System	22-May-07	19-Feb-13	QA
S-ALL-Q-029	03	MintMiner Data File Review	20-Nov-07	19-Dec-12	QA
S-ALL-Q-030	04	Operation of Data Checker for Epic Pro	5-Aug-10	21-Mar-12	QA
S-ALL-Q-034	01	Anonymous Hotline Procedure	5-Aug-10	24-Aug-11	QA
S-ALL-Q-035	02	Data Recall	31-Jan-11	4-Jun-13	QA
S-GB-Q-001	04	Employee Master Signature Log	10-Jan-06	9-Dec-13	QA
S-GB-Q-002	04	Training Record Files Maintained by the QAO	10-Jan-06	9-Dec-13	QA
S-GB-Q-003	03	Data Reduction, Validation, and Reporting	5-Jan-09	7-Jan-14	QA
S-GB-Q-004	04	Laboratory Notebooks and Logbooks	17-Jan-06	9-Dec-13	QA
S-GB-Q-005	04	Precision and Accuracy Measurement and Evaluation	23-Mar-06	9-Dec-13	QA
S-GB-Q-006	04	Data Archiving	27-Apr-06	9-Dec-13	QA
S-GB-Q-007	05	Method of Syringe Technique	9-May-06	9-Dec-13	QA
S-GB-Q-008-	05	Preventative, routine, and non-routine maintenance	9-May-06	7-Jan-14	QA
S-GB-Q-009	04	Common Laboratory Calculations and Statistical Evaluation of Data	11-May-06	29-Nov-12	QA
S-GB-Q-010	04	Estimation of Measurement Uncertainty	15-Feb-07	25-Jul-12	QA
S-GB-Q-011	01	Method Validation and Modification Studies	7-Jun-10	25-Jul-12	QA
S-GB-Q-012	03	Purchasing of Laboratory Supplies	3-Apr-06	13-Mar-14	QA
S-GB-Q-013	03	Receipt and Storage of Laboratory Supplies	21-Mar-06	7-Jan-14	QA
S-GB-Q-016	01	Management of Change	11-May-11	25-Jul-12	QA
S-GB-Q-017	01	Preparation of SOPs	5-May-04	21-Sep-12	QA
S-GB-Q-018	02	Evaluation and Qualification of Vendors	10-Apr-08	8-Jan-14	QA
S-GB-Q-019	01	Software Validation	9-Jun-09	25-Jul-12	QA
S-GB-Q-020	02	Determination of LOD and LOQ	21-Feb-05	7-Jan-14	QA
S-GB-Q-021	02	Proficiency Testing Program	15-Feb-07	7-Jan-14	QA
S-GB-Q-022	02	Internal and External Audits	30-Dec-04	13-Mar-14	QA
S-GB-Q-023	02	MCL Violation Reporting	11-May-09	13-Mar-14	QA
S-GB-Q-024	01	Manual Integration	15-Mar-05	29-Nov-12	QA
S-GB-Q-025	02	Sample Homogenization and Sub-Sampling	16-Nov-06	13-Mar-14	QA
S-GB-Q-026	01	Standard and Reagent Management and Traceability	9-Feb-11	29-Nov-12	QA
S-GB-Q-027	01	Corrective Action / Preventative Action Process	17-Aug-05	19-Feb-13	QA
S-GB-Q-028	02	Monitoring Storage Units	20-Apr-05	19-Feb-13	QA
S-GB-Q-029	01	Document Management	30-Dec-04	19-Feb-13	QA
S-GB-Q-030	01	Support Equipment	17-Jan-05	19-Feb-13	QA
S-GB-Q-031	01	Control Charting and Trend Analysis	9-Feb-11	19-Feb-13	QA
S-GB-Q-032	00	Data Review Process	9-Feb-11	13-May-13	QA
Safety					
S-ALL-S-001	04	Hazard Assessment	1-Jan-05	5-Oct-12	Safety
S-GB-S-001	06	Regulated Soil Handling	5-Jan-06	10-Feb-14	Safety
S-GB-S-002	03	Control of Hazardous Energy Program - Lockout/Tagout	10-Jan-06	21-Sep-12	Safety
S-GB-S-004	03	Rescue Alert System Operation	16-Nov-05	21-Sep-12	Safety
S-GB-S-007	01	Label Destruction	3-Sep-10	4-Oct-13	Safety
S-GB-S-008	00	Air Quality Monitoring and Fume Hood Monitoring	4-Dec-12	10-Dec-12	Safety
Waste					
S-GB-W-001	02	Waste Handling and Management	29-Jul-05	20-Jan-14	Waste
S-GB-W-002	01	Waste Management Training Requirements	11-Feb-09	9-Apr-13	Safety
Equipment					
S-GB-E-001	02	Use and Maintenance of the NANOpure Infinity Water Purification System	28-Mar-06	27-Sep-11	Equipment
S-GB-E-002	03	Operation of Waste Disposal Equipment	21-Sep-07	13-May-13	Equipment
Training					
S-ALL-T-002	04	Learn Center	25-Apr-11	16-Nov-12	Training
Laboratory					
S-GB-L-001	08	Biological Tissue, Plant, and Synthetic Material Preparation	20-Jan-05	6-Feb-14	Laboratory

SOP Number	Rev.	SOP Name	Date of Pub.	Signature Date	Category
S-GB-L-002	03	Small Rodent Handling and Homogenization	20-Jan-05	28-Sep-12	Laboratory
S-GB-L-003	04	The Determination of Lipids in Tissues, Fats, and Plants	20-Jan-05	9-Sep-11	Laboratory
S-GB-L-004	03	Determination of Percent Solids in Tissue Samples	3-Feb-05	9-Sep-11	Laboratory
S-GB-L-005	01	Reagent Water Quality	10-Jan-08	28-Sep-11	Laboratory
S-GB-L-007	00	Cleaning of Equipment Used in the Process of Homogenizing Biological Tissue, Plant, and Synthetic Materials.	19-Jul-11	9-Sep-11	Laboratory

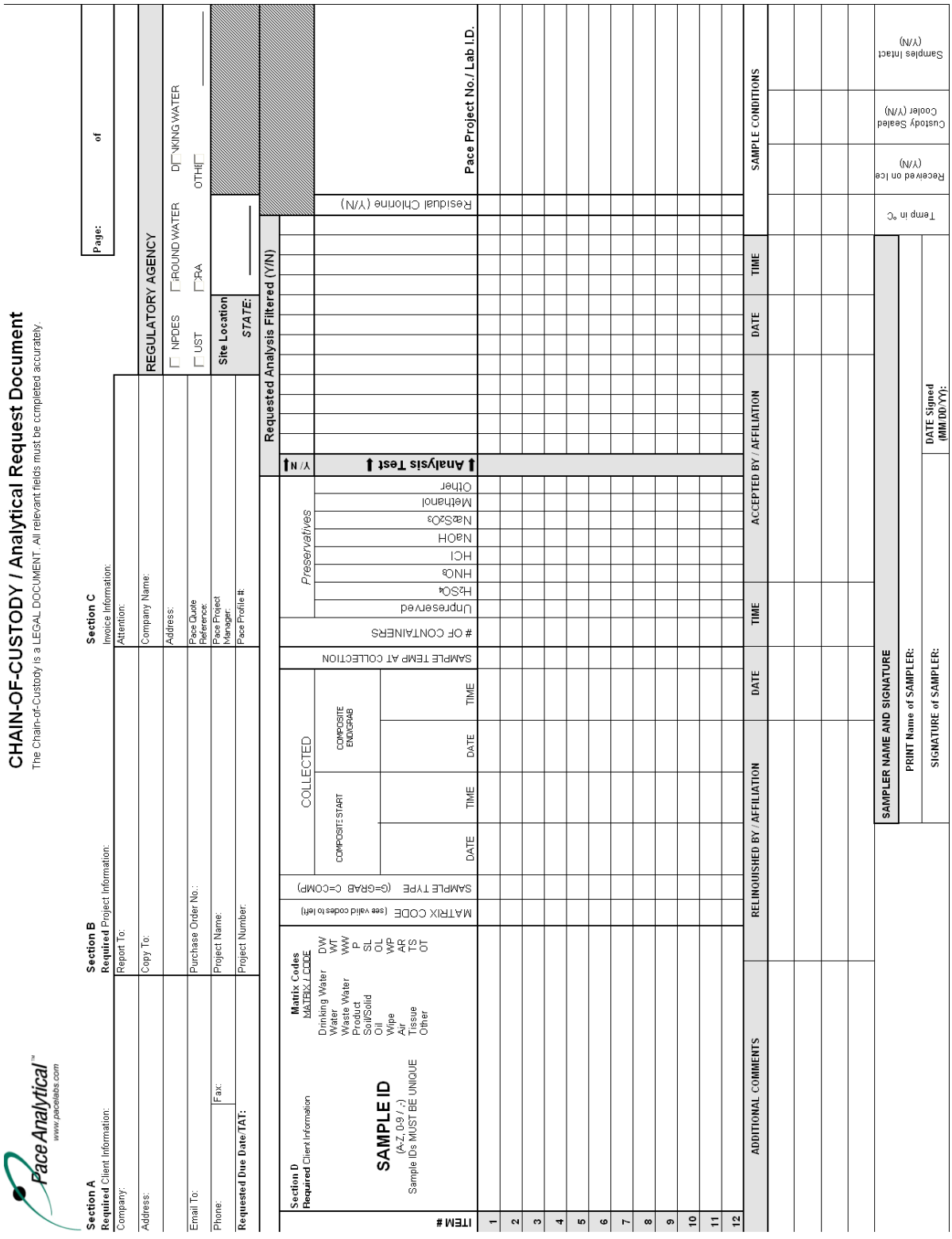
ATTACHMENT VI- LABORATORY CERTIFICATION LIST (CURRENT AS OF ISSUE DATE)

SCOPE AND APPLICATION CERTIFICATES ARE MAINTAINED AND FILED IN THE LOCAL QUALITY DEPARTMENT

Green Bay Laboratory

Accrediting Authority	Program Category	Accrediting Agency	Certification #	Expiration Date
Florida (NELAP)	Biological Tissue	Dept of Health, Bureau of Laboratories	E87948	6/30/2014
Florida (NELAP)	Hazardous Waste - Solid	Dept of Health, Bureau of Laboratories	E87948	6/30/2014
Florida (NELAP)	Waste Water	Dept of Health, Bureau of Laboratories	E87948	6/30/2014
Georgia	Waste Water - NELAP stipulation	Environmental Protection Divison	E87948	6/30/2014
Georgia	Waste Water - NELAP stipulation	Environmental Protection Divison	E87948	6/30/2014
Illinois (NELAP)	Hazardous Waste - Solid	Illinois EPA	200050	8/3/2014
Illinois (NELAP)	Waste Water	Illinois EPA	200050	8/3/2014
Kentucky	UST	Environmental and Public Protection Cabinet	82	6/30/2014
Louisiana (NELAP)	Hazardous Waste - Solid	Department of Environmental Quality	4168	6/30/2014
Louisiana (NELAP)	Waste Water	Department of Environmental Quality	4168	6/30/2014
Louisiana (NELAP)	Biological Tissue	Department of Environmental Quality	4168	6/30/2014
Minnesota	Hazardous Waste	Dept of Health	055-999-334	12/31/2014
Minnesota	Waste Water	Dept of Health	055-999-344	12/31/2014
Minnesota	UST	Department of Health	055-999-344	12/31/2014
New York (NELAP)	Waste Water	Dept of Health	11888	4/1/2015
North Dakota	Hazardous Waste	Dept of Health Chemistry Division	R-150	6/30/2014
North Dakota	Waste Water	Dept of Health Chemistry Division	R-150	6/30/2014
South Carolina	Hazardous Waste	Dept of Health & Environmental Control	83006001	6/30/2014
South Carolina	Waste Water	Dept of Health & Environmental Control	83006001	6/30/2014
US Dept of Agriculture	Foreign Soil Permit	Dept of Agriculture	S-76505	6/4/2016
Wisconsin	Drinking Water	Dept of Natural Resources	405132750	8/31/2014
Wisconsin	Hazardous Waste	Dept of Natural Resources	405132750	8/31/2014
Wisconsin	Waste Water	Dept of Natural Resources	405132750	8/31/2014
Wisconsin	Drinking Water	Dept of Agriculture, Trade & Consumer Protection	105-444	12/31/2014

ATTACHMENT VII- PACE CHAIN-OF-CUSTODY (CURRENT AS OF ISSUE DATE)



ATTACHMENT VIII- METHOD HOLD TIME, CONTAINER AND PRESERVATION GUIDE (CURRENT AS OF ISSUE DATE)

THE HOLDING TIME INDICATED IN THE CHART BELOW IS THE MAXIMUM ALLOWABLE TIME FROM COLLECTION TO EXTRACTION AND/OR ANALYSIS PER THE ANALYTICAL METHOD. FOR METHODS THAT REQUIRE PROCESSING PRIOR TO ANALYSIS, THE HOLDING TIME IS DESIGNATED AS 'PREPARATION HOLDING TIME/ANALYSIS HOLDING TIME'.

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acidity	SM2310B	Water	Plastic/Glass	$\leq 6^oC$	14 Days
Actinides	HASL-300	Water		pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid		None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	$\leq 6^oC$	14 Days
Alkylated PAHs		Water		$\leq 6^oC$; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid		$\leq 10^oC$	1 Year/40 Days
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid		None	180 days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	$\leq 6^oC$; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	$\leq 6^oC$	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	$\leq 6^oC$	28 days
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; $\leq 6^oC$; $Na_2S_2O_3$ if	14 Days (7 Days for aromatics if unpreserved)

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
				Cl present	
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	≤ 6°C	14 Days
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	≤ 6°C; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Solid	8oz Glass	≤ 6°C	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & Pesticides	525.2	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C; Na sulfite if Cl present	14/30 Days
Biomarkers		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Biomarkers		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤ 6°C	48 hours
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	14 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	48 Hours
Carbamates	531.1	Water	Glass	$Na_2S_2O_3$, Monochloro acetic acid pH <3; ≤ 6°C	28 Days
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil	≤ 6°C	48 Hours to filtration
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 6°C	8 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 6°C	8 Hours
Coliform, Fecal	SM9221E	Water	100mL Plastic	≤ 6°C	8 Hours
Coliform, Fecal	SM9221E	Solid	100mL Plastic	≤ 6°C	24 Hours
Coliform, Total	SM9222B	Water	100mL Plastic	≤ 6°C	8 Hours
Coliform, Total	SM9221B	Solid	100mL Plastic	≤ 6°C	8 Hours

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Coliform, Total and E. coli	SM9223B	Drinking Water	100mL Plastic	≤ 10°C	30 Hours after collection
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	24 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	180 Days
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/ 9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6°C; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present-applies to SM4500CN only)
Diesel Range Organics- Alaska DRO	AK102	Solid	8oz Glass	≤ 6°C	14/40 Days
Diesel Range Organics- Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10°C	1 Year if frozen/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; ≤ 6°C	14/40 Days; 7 Days from collection to extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6°C	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6°C; pH <2 HCl	7/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ 6°C	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 year
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	≤ 6°C	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if	30/45 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
				Cl present	
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6°C	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10°C	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	30/45 Days
Diquat/Paraquat	549.2	Water	Amber Plastic	≤ 6°C; $Na_2S_2O_3$	7/21 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Endothall	548.1	Water	Amber Glass	≤ 6°C; $Na_2S_2O_3$	7/14 Days
Enterococci	EPA 1600	Water	100mL Plastic	≤ 6°C	8 Hours
Explosives	8330/8332	Water	1L Amber Glass	≤ 6°C	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Fecal Streptococci	SM9230B	Water	100mL Plastic	≤ 6°C	8 Hours
Ferrous Iron	SN3500Fe-D; Hach 8146	Water	Glass	None	Immediate
Flashpoint/Ignitability	1010	Liquid	Plastic/Glass	None	28 Days
Florida PRO	FL PRO DEP (11/1/95)	Liquid	Glass, PTFE lined cap	≤ 6°C; pH <2 H_2SO_4 or HCl	7/40 Days
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics-Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics-Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; ≤ 6°C	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	≤ 6°C; packed jars with no headspace	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	≤ 6°C in MeOH	21 Days
Glyphosate	547	Water	Glass	≤ 6°C; $Na_2S_2O_3$	14 Days (18 Months frozen)
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤ 6°C	14/7 Days if extracts stored ≤ 6°C or 14/14 Days if extracts stored at ≤ -10°C
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Heterotrophic Plate Count (SPC/HPC)	SM9215B	Water	100mL Plastic	≤ 6°C	24 Hours
Heterotrophic Plate Count (SPC/HPC)	SimPlate	Water	100mL Plastic	≤ 6°C	8 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/SM3500Cr-B, C, D	Water	Plastic/Glass	≤ 6°C	24 Hours (see note 4)
Hexavalent Chromium	7196/218.6/SM3500Cr-B, C, D	Water	Plastic/Glass	Ammonium Buffer pH 9.3-9.7	28 Days (see note 4)
Hexavalent Chromium	218.6/218.7	Drinking Water	Plastic/Glass	Ammonium Buffer pH >8	14 Days (see note 4)
Hexavalent Chromium	7196 (with 3060A)	Solid		≤ 6°C	24 Hours after extraction
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
Lead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months
Lipids	Pace Lipids	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid	Glass	None	28 Days
Mercury, Low-Level	1631E	Water	Fluoropolyme	12N HCl or	48 Hours for

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
			r bottles (Glass if Hg is only analyte being tested)	BrCl	preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	$\leq$ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	$\leq$ 6°C	28 Days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	$\leq$ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	180 Days
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	180 Days
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	$\leq$ -10°C	180 Days if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
Methanol, Ethanol	8015 modified	Water	40mL vials	$\leq$ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	$\leq$ 6°C	14 Days
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq$ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	$\leq$ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq$ 6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	$\leq$ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	$\leq$ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; $\leq$ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	$\leq$ 6°C	48 Hours

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	14 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	48 Hours
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	28 Days
PBDEs	1614	Water	1L Amber Glass	≤ 6°C	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6°C	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10°C	1 Year/1 Year
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass		Pest: 7/40 Days; PCB: 1 Year/1 Year
PCBs, Pesticides (OC), Herbicides	508.1	Water	Glass	Na2SO3; pH<2 HCl; ≤ 6°C	14/30 Days
Perchlorate	331	Water	Plastic/Glass	>0-6°C	28 Days
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	≤ -10°C	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; ≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	≤ 6°C	1 Year/1 Year

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/1 Year
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10°C	1 Year/1 Year
Oil Range Organics- ORO					
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particulates	PM-10	Air	Filters	None	180 Days
Permanent Gases	EPA 3C	Air	Summa Canister	None	14 Days
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	Filter; ≤ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/40 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid			
Residual Range Organics- Alaska RRO	AK103	Solid	8oz Glass	≤ 6°C	14/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Saturated Hydrocarbons		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Saturated Hydrocarbons		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6°C	28 Days
Stationary Source Dioxins and Furans	EPA 23	Air	XAD Trap	None	30/45 Days
Stationary Source Mercury	EPA 101	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	180 Days, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	180 Days
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	180 Days
Sulfate	SM4500SO4/9036/9038/375.2/ASTM D516	Water	Plastic/Glass	≤ 6°C	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤ 6°C	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black	Solid	Glass	≤ 6°C	14 Days
Total Organic Halogen (TOX)	SM5320/9020/9021	Water	Glass; no headspace	≤ 6°C	14 Days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Uranium	908.0/ASTM D5174-	Water	Plastic/Glass	pH<2 HCl	180 days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
	97				
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days preserved
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Solid	4-8oz Glass Jar	≤ 6°C; packed jars with no headspace	7/28 Days
Volatiles	TO-14	Air	Summa Canister	None	30 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	48 Hours
Volatiles	TO-15	Air	Summa Canister	None	30 Days
Volatiles	TO-18/8260	Air	Tedlar Bag or equivalent	None	72 Hours
Volatiles	8260	Solid	5035 vial kit	See note 1	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6°C; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days
UCMR3 Metals	200.8	Water	Plastic or glass	pH<2 HNO_3	28 Days
UCMR3 Hexavalent Chromium	218.7	Water	HDPE or propylene	Na_2CO_3/$NaHCO_3$/$(NH_4)_2SO_4$; pH>8	14 Days
UCMR3 Chlorate	300.1	Water	Plastic or glass	EDA	28 Days
UCMR3 Hormones	539	Water	Amber glass	$Na_2S_2O_3$, 2-mercaptopyridine-1-oxide, sodium salt	28 Days
UCMR3 Perfluorinated	537	Water	Polypropylene	Trizma	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Compounds					
UCMR3 Volatiles	524.3	Water	40 mL amber glass vials	Ascorbic acid. Maleic acid pH~2	14 Days
UCMR3 1, 4 Dioxane	522	Water	Glass	Na_2SO_3, $NaHSO_4$; pH<4	28 Days
UV254	SM5910B	Water	Glass	$\leq 6^oC$	48 Hours

[1] **5035/5035A Note**: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4°C, **and** one vial methanol preserved at ≤6°C **and** one container of unpreserved sample stored at ≤6°C.

[2] Method 524.2 lists ascorbic acid as the preservative when residual chlorine is suspected, unless gases or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

[3] Methods 9315 and 9320 both state that if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation/analysis.

[4] The holding time for hexavalent chromium may be extended by the addition of the ammonium buffer listed in EPA 218.6 per the 2012 EPA Method Update Rule.Although Method 218.6 stipulates a different pH range (9.0 to 9.5) for buffering, this method requirement was modified in the Method Update Rule to a pH range of 9.3 to 9.7.For non-potable waters, adjust the pH of the sample to 9.3 to 9.7 during collection with the method required ammonium sulfate bufferto extend the holding time to 28 days. For potable waters, addition of the buffer during collection will extend the holding time for 14 days per EPA 218.7 and the EPA UCMR3 program.

ATTACHMENT IX- NELAC CERTIFICATE

Florida HEALTH

State of Florida

Department of Health, Bureau of Public Health Laboratories

This is to certify that

E87948

PACE ANALYTICAL SERVICES, INC - GREEN BAY #1
1241 BELLEVUE STREET
GREEN BAY, WI 54302

has complied with Florida Administrative Code 64E-1,
for the examination of environmental samples in the following categories

NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS, BIOLOGICAL TISSUE - EXTRACTABLE ORGANICS, BIOLOGICAL TISSUE - GENERAL CHEMISTRY, BIOLOGICAL TISSUE - METALS, BIOLOGICAL TISSUE - PESTICIDES-HERBICIDES-PCB'S

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2013 Expiration Date: June 30, 2014

Victor Johnson

Victor Johnson, Director
Division of Emergency Preparedness and Community Support
DH Form 1697, 7/04
NON-TRANSFERABLE E87948-22-07/01/2013
Supersedes all previously issued certificates

NELAP RECOGNIZED ACCREDITATION BODY

GREAT SEAL OF THE STATE OF FLORIDA IN GOD WE TRUST

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-22, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	3/19/2012
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2,4-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	5/15/2009
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3,5-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87948-22, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	4/1/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	5/15/2009
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Hexanone	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Rick Scott
Governor

Florida HEALTH

John H. Armstrong, MD, FACS
State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-22, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	4/1/2005
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	4/1/2005
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthene	EPA 625	Extractable Organics	NELAP	4/1/2005
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthylene	EPA 625	Extractable Organics	NELAP	4/1/2005
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acetone	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetophenone	EPA 625	Extractable Organics	NELAP	4/1/2005
Acetophenone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acidity, as CaCO3	EPA 305.1	General Chemistry	NELAP	4/1/2005
Acidity, as CaCO3	SM 2310 B	General Chemistry	NELAP	5/22/2007
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	5/15/2009
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acrylonitrile	EPA 624	Volatile Organics	NELAP	5/15/2009
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2013** **Expiration Date: 6/30/2014**

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Laboratory Scope of Accreditation

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Attachment to Certificate #: E87948-22, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Alkalinity as CaCO3	EPA 310.2	General Chemistry	NELAP	4/1/2005
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aluminum	EPA 6010	Metals	NELAP	4/1/2005
Aluminum	EPA 6020	Metals	NELAP	4/1/2005
Ammonia as N	EPA 350.1	General Chemistry	NELAP	4/1/2005
Aniline	EPA 625	Extractable Organics	NELAP	4/1/2005
Aniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
Anthracene	EPA 625	Extractable Organics	NELAP	4/1/2005
Anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Antimony	EPA 6010	Metals	NELAP	4/1/2005
Antimony	EPA 6020	Metals	NELAP	4/1/2005
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Arsenic	EPA 6010	Metals	NELAP	4/1/2005
Arsenic	EPA 6020	Metals	NELAP	4/1/2005
Barium	EPA 6010	Metals	NELAP	4/1/2005
Barium	EPA 6020	Metals	NELAP	4/1/2005
Benzene	EPA 602	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 624	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Benzidine	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2005

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Rick Scott
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Florida HEALTH

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State Surgeon General & Secretary

Laboratory Scope of Accreditation

Attachment to Certificate #: E87948-22, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2005
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzoic acid	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Beryllium	EPA 6010	Metals	NELAP	4/1/2005
Beryllium	EPA 6020	Metals	NELAP	4/1/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	4/1/2005
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	4/1/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/1/2005
Boron	EPA 6010	Metals	NELAP	4/1/2005
Boron	EPA 6020	Metals	NELAP	4/1/2005
Bromide	EPA 300.0	General Chemistry	NELAP	4/1/2005
Bromide	EPA 9056	General Chemistry	NELAP	4/1/2005
Bromobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromoform	EPA 624	Volatile Organics	NELAP	5/15/2009
Bromoform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Cadmium	EPA 6010	Metals	NELAP	4/1/2005
Cadmium	EPA 6020	Metals	NELAP	4/1/2005
Calcium	EPA 6010	Metals	NELAP	4/1/2005
Calcium	EPA 6020	Metals	NELAP	4/1/2005

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbazole	EPA 8270	Extractable Organics	NELAP	4/1/2005
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	5/15/2009
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	5/15/2009
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	4/1/2005
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	4/1/2005
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Chloride	EPA 300.0	General Chemistry	NELAP	4/1/2005
Chloride	EPA 9056	General Chemistry	NELAP	4/1/2005
Chlorobenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Chloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroform	EPA 624	Volatile Organics	NELAP	5/15/2009
Chloroform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroprene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chromium	EPA 6010	Metals	NELAP	4/1/2005
Chromium	EPA 6020	Metals	NELAP	4/1/2005
Chromium VI	SM 3500-Cr B (20th/21st Ed.)/UV-VIS	General Chemistry	NELAP	5/30/2007
Chrysene	EPA 625	Extractable Organics	NELAP	4/1/2005
Chrysene	EPA 8270	Extractable Organics	NELAP	4/1/2005
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	5/15/2009
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Cobalt	EPA 6010	Metals	NELAP	4/1/2005
Cobalt	EPA 6020	Metals	NELAP	4/1/2005
Conductivity	EPA 120.1	General Chemistry	NELAP	4/1/2005
Copper	EPA 6010	Metals	NELAP	4/1/2005
Copper	EPA 6020	Metals	NELAP	4/1/2005
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	4/1/2005
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009

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Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibromomethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/1/2005
Diethyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	4/1/2005
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Ethanol	EPA 8015	Volatile Organics	NELAP	5/15/2009
Ethanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 602	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 624	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl-t-butylether (ETBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Fluoranthene	EPA 625	Extractable Organics	NELAP	4/1/2005
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Fluorene	EPA 625	Extractable Organics	NELAP	4/1/2005
Fluorene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Fluoride	EPA 300.0	General Chemistry	NELAP	4/1/2005
Fluoride	EPA 9056	General Chemistry	NELAP	4/1/2005
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	5/15/2009
Gasoline range organics (GRO)	WI-GRO	Volatile Organics	NELAP	5/15/2009
Hardness	SM 2340 B	General Chemistry	NELAP	4/1/2005
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachloroethane	EPA 625	Extractable Organics	NELAP	4/1/2005
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/1/2005
Ignitability	EPA 1010	General Chemistry	NELAP	4/1/2005
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Iron	EPA 6010	Metals	NELAP	4/1/2005
Iron	EPA 6020	Metals	NELAP	4/1/2005
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isophorone	EPA 625	Extractable Organics	NELAP	4/1/2005
Isophorone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Isopropyl alcohol (2-Propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	4/1/2005
Lead	EPA 6010	Metals	NELAP	4/1/2005
Lead	EPA 6020	Metals	NELAP	4/1/2005
Lithium	EPA 6020	Metals	NELAP	5/15/2009
m/p-Xylenes	EPA 8021	Volatile Organics	NELAP	5/15/2009
m/p-Xylenes	EPA 8260	Volatile Organics	NELAP	5/15/2009
Magnesium	EPA 6010	Metals	NELAP	4/1/2005
Magnesium	EPA 6020	Metals	NELAP	4/1/2005
Manganese	EPA 6010	Metals	NELAP	4/1/2005
Manganese	EPA 6020	Metals	NELAP	4/1/2005

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Laboratory Scope of Accreditation

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Pace Analytical Services, Inc - Green Bay #1
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Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Mercury	EPA 1631	Metals	NELAP	5/30/2007
Mercury	EPA 6020	Metals	NELAP	3/19/2012
Mercury	EPA 7470	Metals	NELAP	4/1/2005
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methanol	EPA 8015	Volatile Organics	NELAP	5/15/2009
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	5/15/2009
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	5/15/2009
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methylene chloride	EPA 624	Volatile Organics	NELAP	5/15/2009
Methylene chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Molybdenum	EPA 6010	Metals	NELAP	4/1/2005
Molybdenum	EPA 6020	Metals	NELAP	4/1/2005
Naphthalene	EPA 625	Extractable Organics	NELAP	4/1/2005
Naphthalene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Nickel	EPA 6010	Metals	NELAP	4/1/2005
Nickel	EPA 6020	Metals	NELAP	4/1/2005
Nitrate	EPA 9056	General Chemistry	NELAP	4/1/2005
Nitrate as N	EPA 300.0	General Chemistry	NELAP	4/1/2005
Nitrite	EPA 9056	General Chemistry	NELAP	4/1/2005
Nitrite as N	EPA 300.0	General Chemistry	NELAP	4/1/2005
Nitrobenzene	EPA 625	Extractable Organics	NELAP	4/1/2005
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	4/1/2005
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	4/1/2005
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Propanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	4/1/2005
o-Xylene	EPA 8021	Volatile Organics	NELAP	5/15/2009
o-Xylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	4/1/2005
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
pH	EPA 9040	General Chemistry	NELAP	4/1/2005
pH	SM 4500-H+-B	General Chemistry	NELAP	5/22/2007
Phenanthrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phenol	EPA 625	Extractable Organics	NELAP	4/1/2005
Phenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	4/1/2005
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Potassium	EPA 6010	Metals	NELAP	4/1/2005
Potassium	EPA 6020	Metals	NELAP	4/1/2005
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Pyrene	EPA 625	Extractable Organics	NELAP	4/1/2005
Pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Pyridine	EPA 625	Extractable Organics	NELAP	4/1/2005
Pyridine	EPA 8270	Extractable Organics	NELAP	4/1/2005
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	5/30/2007
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	5/30/2007
Residue-total	SM 2540 B	General Chemistry	NELAP	5/30/2007
Residue-volatile	EPA 160.4	General Chemistry	NELAP	4/1/2005
Residue-volatile	SM 2540 E	General Chemistry	NELAP	5/30/2007
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Selenium	EPA 6010	Metals	NELAP	4/1/2005
Selenium	EPA 6020	Metals	NELAP	4/1/2005
Silicon	EPA 6020	Metals	NELAP	3/19/2012
Silver	EPA 6010	Metals	NELAP	4/1/2005
Silver	EPA 6020	Metals	NELAP	4/1/2005
Sodium	EPA 6010	Metals	NELAP	4/1/2005

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Sodium	EPA 6020	Metals	NELAP	4/1/2005
Strontium	EPA 6020	Metals	NELAP	5/30/2007
Styrene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Sulfate	EPA 300.0	General Chemistry	NELAP	4/1/2005
Sulfate	EPA 9056	General Chemistry	NELAP	4/1/2005
Sulfide	SM 4500-S F (19th/20th/21st Ed.)/TITR	General Chemistry	NELAP	1/30/2008
T-amylmethylether (TAME)	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	5/15/2009
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Thallium	EPA 6010	Metals	NELAP	4/1/2005
Thallium	EPA 6020	Metals	NELAP	4/1/2005
Tin	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6020	Metals	NELAP	5/30/2007
Toluene	EPA 602	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 624	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Total cyanide	EPA 335.4	General Chemistry	NELAP	4/1/2005
Total cyanide	EPA 9012	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	4/1/2005
Total organic carbon	EPA 9060	General Chemistry	NELAP	6/24/2011
Total organic carbon	SM 5310 C	General Chemistry	NELAP	6/24/2011
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	5/15/2009
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-Nonachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	5/15/2009
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009

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State Surgeon General & Secretary

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Attachment to Certificate #: E87948-22, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	5/15/2009
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Turbidity	SM 2130 B	General Chemistry	NELAP	4/1/2005
Uranium	EPA 6020	Metals	NELAP	3/19/2012
Vanadium	EPA 6010	Metals	NELAP	4/1/2005
Vanadium	EPA 6020	Metals	NELAP	4/1/2005
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Vinyl chloride	EPA 624	Volatile Organics	NELAP	5/15/2009
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 602	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 624	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Zinc	EPA 6010	Metals	NELAP	4/1/2005
Zinc	EPA 6020	Metals	NELAP	4/1/2005
Zirconium	EPA 6020	Metals	NELAP	3/19/2012

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	3/19/2012
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2,4-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3,5-Trimethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	5/15/2009
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005

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Attachment to Certificate #: E87948-22, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Hexanone	EPA 8260	Volatile Organics	NELAP	5/15/2009
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	4/1/2005
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	5/15/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acetone	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acetophenone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aluminum	EPA 6010	Metals	NELAP	4/1/2005
Aluminum	EPA 6020	Metals	NELAP	4/1/2005
Ammonia as N	EPA 350.1	General Chemistry	NELAP	4/1/2005
Aniline	EPA 8270	Extractable Organics	NELAP	4/1/2005
Anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Antimony	EPA 6010	Metals	NELAP	4/1/2005
Antimony	EPA 6020	Metals	NELAP	4/1/2005
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Arsenic	EPA 6010	Metals	NELAP	4/1/2005
Arsenic	EPA 6020	Metals	NELAP	4/1/2005
Barium	EPA 6010	Metals	NELAP	4/1/2005
Barium	EPA 6020	Metals	NELAP	4/1/2005
Benzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Benzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Benzidine	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzoic acid	EPA 8270	Extractable Organics	NELAP	4/1/2005
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Beryllium	EPA 6010	Metals	NELAP	4/1/2005
Beryllium	EPA 6020	Metals	NELAP	4/1/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/1/2005

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/1/2005
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/1/2005
Boron	EPA 6010	Metals	NELAP	4/1/2005
Boron	EPA 6020	Metals	NELAP	4/1/2005
Bromobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Bromoform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Cadmium	EPA 6010	Metals	NELAP	4/1/2005
Cadmium	EPA 6020	Metals	NELAP	4/1/2005
Calcium	EPA 6010	Metals	NELAP	4/1/2005
Calcium	EPA 6020	Metals	NELAP	4/1/2005
Carbazole	EPA 8270	Extractable Organics	NELAP	4/1/2005
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	5/15/2009
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroform	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chloroprene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Chromium	EPA 6010	Metals	NELAP	4/1/2005
Chromium	EPA 6020	Metals	NELAP	4/1/2005
Chrysene	EPA 8270	Extractable Organics	NELAP	4/1/2005
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Cobalt	EPA 6010	Metals	NELAP	4/1/2005
Cobalt	EPA 6020	Metals	NELAP	4/1/2005
Copper	EPA 6010	Metals	NELAP	4/1/2005
Copper	EPA 6020	Metals	NELAP	4/1/2005
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dibromomethane	EPA 8260	Volatile Organics	NELAP	5/15/2009

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Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	4/1/2005
Diethyl ether	EPA 8260	Volatile Organics	NELAP	5/15/2009
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/1/2005
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Ethanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Ethyl-t-butylether (ETBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Fluorene	EPA 8270	Extractable Organics	NELAP	4/1/2005
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	5/15/2009
Gasoline range organics (GRO)	WI-GRO	Extractable Organics	NELAP	5/15/2009
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/1/2005
Ignitability	EPA 1010	General Chemistry	NELAP	4/1/2005
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Iron	EPA 6010	Metals	NELAP	4/1/2005
Iron	EPA 6020	Metals	NELAP	4/1/2005
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isophorone	EPA 8270	Extractable Organics	NELAP	4/1/2005
Isopropyl alcohol (2-Propanol)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	4/1/2005
Lead	EPA 6010	Metals	NELAP	4/1/2005
Lead	EPA 6020	Metals	NELAP	4/1/2005
Lithium	EPA 6020	Metals	NELAP	5/15/2009
Magnesium	EPA 6010	Metals	NELAP	4/1/2005
Magnesium	EPA 6020	Metals	NELAP	4/1/2005
Manganese	EPA 6010	Metals	NELAP	4/1/2005
Manganese	EPA 6020	Metals	NELAP	4/1/2005
Mercury	EPA 6020	Metals	NELAP	3/19/2012
Mercury	EPA 7471	Metals	NELAP	4/1/2005
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Methylene chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Molybdenum	EPA 6010	Metals	NELAP	4/1/2005
Molybdenum	EPA 6020	Metals	NELAP	4/1/2005
Naphthalene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Nickel	EPA 6010	Metals	NELAP	4/1/2005
Nickel	EPA 6020	Metals	NELAP	4/1/2005
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/1/2005

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State Surgeon General & Secretary

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87948-22, expiration date June 30, 2014. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/1/2005
n-Propanol	EPA 8260	Volatile Organics	NELAP	5/15/2009
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	4/1/2005
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	4/1/2005
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Percent (%) moisture	ASTM D2974-87	General Chemistry	NELAP	5/15/2009
pH	EPA 9040	General Chemistry	NELAP	4/1/2005
pH	EPA 9045	General Chemistry	NELAP	4/1/2005
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phenol	EPA 8270	Extractable Organics	NELAP	4/1/2005
Phosphorus	EPA 6020	Metals	NELAP	3/19/2012
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	4/1/2005
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Potassium	EPA 6010	Metals	NELAP	4/1/2005
Potassium	EPA 6020	Metals	NELAP	4/1/2005
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Pyrene	EPA 8270	Extractable Organics	NELAP	4/1/2005
Pyridine	EPA 8270	Extractable Organics	NELAP	4/1/2005
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Selenium	EPA 6010	Metals	NELAP	4/1/2005
Selenium	EPA 6020	Metals	NELAP	4/1/2005
Silver	EPA 6010	Metals	NELAP	4/1/2005
Silver	EPA 6020	Metals	NELAP	4/1/2005
Sodium	EPA 6010	Metals	NELAP	4/1/2005
Sodium	EPA 6020	Metals	NELAP	4/1/2005
Strontium	EPA 6020	Metals	NELAP	5/30/2007
Styrene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	4/1/2005
T-amylmethylether (TAME)	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/15/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Thallium	EPA 6010	Metals	NELAP	4/1/2005

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Green Bay, WI 54302

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Thallium	EPA 6020	Metals	NELAP	4/1/2005
Tin	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6010	Metals	NELAP	4/1/2005
Titanium	EPA 6020	Metals	NELAP	5/30/2007
Toluene	EPA 8021	Volatile Organics	NELAP	5/15/2009
Toluene	EPA 8260	Volatile Organics	NELAP	5/15/2009
Total cyanide	EPA 9012	General Chemistry	NELAP	4/1/2005
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	4/1/2005
Total nitrogen	TKN + Total nitrate-nitrite	General Chemistry	NELAP	4/1/2005
Total organic carbon	EPA 9060	General Chemistry	NELAP	5/30/2007
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	4/1/2005
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	5/15/2009
trans-Nonachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	5/15/2009
Uranium	EPA 6020	Metals	NELAP	3/19/2012
Vanadium	EPA 6010	Metals	NELAP	4/1/2005
Vanadium	EPA 6020	Metals	NELAP	4/1/2005
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	5/15/2009
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8021	Volatile Organics	NELAP	5/15/2009
Xylene (total)	EPA 8260	Volatile Organics	NELAP	5/15/2009
Zinc	EPA 6010	Metals	NELAP	4/1/2005
Zinc	EPA 6020	Metals	NELAP	4/1/2005

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State Surgeon General & Secretary

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	5/30/2007
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	5/30/2007
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	5/30/2007
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	5/30/2007
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
Acenaphthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aluminum	EPA 6020	Metals	NELAP	5/30/2007

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State Surgeon General & Secretary

Laboratory Scope of Accreditation

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State Laboratory ID: E87948 **EPA Lab Code: WI01103** **(920) 469-2436**

E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Anthracene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Antimony	EPA 6020	Metals	NELAP	5/30/2007
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Arsenic	EPA 6020	Metals	NELAP	5/30/2007
Barium	EPA 6020	Metals	NELAP	5/30/2007
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Beryllium	EPA 6020	Metals	NELAP	5/30/2007
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	5/30/2007
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	5/30/2007
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	5/30/2007
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	5/30/2007
Boron	EPA 6020	Metals	NELAP	5/30/2007
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Cadmium	EPA 6020	Metals	NELAP	5/30/2007
Calcium	EPA 6020	Metals	NELAP	5/30/2007
Carbazole	EPA 8270	Extractable Organics	NELAP	5/30/2007
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Chromium	EPA 6020	Metals	NELAP	5/30/2007
Chrysene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Cobalt	EPA 6020	Metals	NELAP	5/30/2007
Copper	EPA 6020	Metals	NELAP	5/30/2007
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	5/30/2007
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007

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Florida HEALTH

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State Surgeon General & Secretary

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E87948
Pace Analytical Services, Inc - Green Bay #1
1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	5/30/2007
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Fluoranthene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Fluorene	EPA 8270	Extractable Organics	NELAP	5/30/2007
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	5/30/2007
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Iron	EPA 6020	Metals	NELAP	5/30/2007
Isophorone	EPA 8270	Extractable Organics	NELAP	5/30/2007
Lead	EPA 6020	Metals	NELAP	5/30/2007
Lithium	EPA 6020	Metals	NELAP	5/15/2009
Magnesium	EPA 6020	Metals	NELAP	5/30/2007
Manganese	EPA 6020	Metals	NELAP	5/30/2007
Mercury	EPA 245.6	General Chemistry	NELAP	5/30/2007
Mercury	EPA 6020	Metals	NELAP	3/19/2012
Mercury	EPA 7471	Metals	NELAP	5/30/2007
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Mirex	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Molybdenum	EPA 6020	Metals	NELAP	5/30/2007
Naphthalene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Nickel	EPA 6020	Metals	NELAP	5/30/2007
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	5/30/2007

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1241 Bellevue Street
Green Bay, WI 54302

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	5/30/2007
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	5/30/2007
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
Phenanthrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Phenol	EPA 8270	Extractable Organics	NELAP	5/30/2007
Potassium	EPA 6020	Metals	NELAP	5/30/2007
Pyrene	EPA 8270	Extractable Organics	NELAP	5/30/2007
Selenium	EPA 6020	Metals	NELAP	5/30/2007
Silver	EPA 6020	Metals	NELAP	5/30/2007
Sodium	EPA 6020	Metals	NELAP	5/30/2007
Strontium	EPA 6020	Metals	NELAP	5/30/2007
Thallium	EPA 6020	Metals	NELAP	5/30/2007
Titanium	EPA 6020	Metals	NELAP	5/30/2007
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
trans-Nonachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	5/30/2007
Vanadium	EPA 6020	Metals	NELAP	5/30/2007
Zinc	EPA 6020	Metals	NELAP	5/30/2007

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Pace Analytical Services, Inc.
1241 Bellevue Street Suite 9
Green Bay, WI 54302
Phone: 920 469-2436
Fax: 920 469-8827

STANDARD OPERATING PROCEDURE

Biochemical Oxygen Demand

Reference Method: SM 5210B-11

SOP NUMBER:	S-GB-I-044-REV.04
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-044-REV.03

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 04/03/14

Kate Grams, Laboratory Quality Manager — Date: 4/3/14

Chad Rusch, Department Manager — Date: 4/3/14

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

This Standard Operating Procedure (SOP) describes the analyses of Biochemical Oxygen Demand (BOD) and carbonaceous BOD (cBOD) in wastewaters, effluents, polluted waters, dissolved, sludges, and soils based on Standard Methods 5210B-01.

2. Summary of Method

BOD is determined by measuring the oxygen utilized during a 5-day incubation period at 20°C in the dark. Dissolved oxygen (DO) is measured initially and after incubation, and the BOD is calculated from the difference between the initial and final DO readings. CBOD is determined by the same procedure, but with the addition of a nitrification inhibitor.

3. Scope and Application

3.1 This procedure is restricted to use by, or under the supervision of, analysts experienced with dissolved oxygen measurements. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.2 This SOP is applies to the analyses of Biochemical Oxygen Demand (BOD) and carbonaceous BOD (cBOD).

4. Applicable Matrices

4.1 This SOP is applicable to the analyses of Biochemical Oxygen Demand (BOD) and carbonaceous BOD (cBOD) in wastewaters, effluents, polluted waters, dissolved, sludges, and soils.

5. Limits of Detection and Quantitation

5.1 The minimum BOD and cBOD Reporting Limit is 2 mg/L.

5.2 The Reporting Limit reported is 2 mg/L multiplied by the dilution of the lowest dilution level that had at least 1.0 mg/L of DO depletion and 2.0 mg/L DO remaining.

6. Interferences

6.1 Free chlorine will cause interference and must be removed by adding sodium sulfite (Na_2SO_3) to the samples.

6.2 Samples containing other interfering toxic substances, such as toxic metals, often require special study and treatment.

6.3 pH values outside the range of 6.0 to 8.0 will cause unreliable results. To correct for this, use sulfuric acid or sodium hydroxide to bring the pH into the proper range of 7.0 to 7.2.

6.4 The temperature of the samples should be 20 °C ±1 °C when performing the analysis.

7. Sample Collection, Preservation, Shipment and Storage

7.1 **Sample Preservation** – To minimize reduction of BOD/cBOD, samples must be stored at or below 4 °C from the time of collection. Chemical preservatives must not be used.

7.2 **Holding Time** – Analysis should be started as soon as possible but not to exceed 48 hours. For samples from the state of Minnesota, analysis should be started as soon as possible but not to exceed 24 hours and 29 minutes.

8. Definitions

Definitions can be found in Section 10 of the most recent version of the Pace Analytical Services, Inc. Quality Manual.

9. Equipment and Supplies

9.1 Incubation Bottles – 300 mL capacity glass bottles with ground glass or plastic stoppers and water seal caps. Bottles must be thoroughly cleaned and contain no soap residue before use.

9.2 pH specific ion meter and buffers. See most current version of Measurement of pH in Water, Soil, and Waste and Addendum (S-GB-ALL-I-15) for instructions.

9.3 Dissolved oxygen meter: YSI Model 5000 with YSI 5010 stirring probe (or equivalent).

9.4 YSI 5906 Membrane cap kit

9.5 Adjustable pipetter, various sized wide bore serological pipettes, pipette bulb.

9.6 50, 100, and 250 mL graduated cylinders.

9.7 Air incubator – thermostatically controlled at 20 ± 1 °C without any light.

9.8 Water bath set at 20 °C.

9.9 12 L glass aspirator bottles.

9.10 Glass or Polypropylene Carboy, Nalgene or equivalent.

9.11 Class A1000 mL volumetric flask.

9.12 Glass microfiber filters (Fisher G6 Cat.# 09-804-90A – or equivalent) for use in dissolved BOD.

9.13 Analytical Balance capable of reading to 0.1 grams.

9.14 Starch Indicator Paper, Precision Labs VWR cat # 60799-008

9.15 Current Barometric Pressure reading from NOAA website as reported by Green Bay airport and corrected to 594 ft above sea level and expressed in mmHg.

9.16 Calibrated Thermometer capable of reading in 0.5 °C increments.

9.17 Lab Solutions Powder detergent (or equivalent).

10. Reagents and Standards

10.1 Phosphate Buffer Solution: Dissolve 8.5 g potassium phosphate monobasic (KH_2PO_4), 21.75 g potassium phosphate dibasic (K_2HPO_4), 33.4 g sodium phosphate dibasic ($Na_2HPO_4 \cdot 7H_2O$), and 1.7 g ammonium chloride (NH_4Cl) in 500 mL distilled water and dilute to 1000 mL. Store in refrigerator. The pH should be 7.2 without further adjustment. Discard if biological growth is present. Alternatively, use a commercially prepared phosphate buffer - Fisher#: SP341-1.

10.2 Magnesium Sulfate: Dissolve 22.5 g magnesium sulfate ($MgSO_4 \cdot 7H_2O$) in distilled water and dilute to 1000 mL. Alternatively, use commercially prepared magnesium sulfate - Fisher#: SM109-1.

10.3 Calcium Chloride: Dissolve 27.5 g anhydrous calcium chloride ($CaCl_2$) in distilled water and dilute to 1000 mL. Alternatively, use commercially prepared calcium chloride - VWR#: VW3308-1.

10.4 Ferric Chloride: Dissolve 0.25 g ferric chloride ($FeCl_3 \cdot 6H_2O$) in distilled water and dilute to 1000 mL. Alternatively, use commercially prepared ferric chloride - VWR#: VW3318-1.

10.5 Sodium Sulfite, anhydrous (Na_2SO_3)

10.6 Sodium Sulfite Solution: Dissolve 1.575g Na_2SO_4 in distilled water and dilute to 1000mL. This solution is not stable: prepare daily.

10.7 Potassium Iodide, anhydrous (KI)

10.8 Potassium Iodide Solution: Used in determining amount of Sodium Sulfite Solution used in de-chlorination. Dissolve 10 g of potassium iodide in distilled water and dilute to 100mL.

10.9 Glucose/Glutamic Acid Standard (198 ± 30.5 mg/l): Purchase RICCA Glucose-Glutamic Acid Solution – Fisher Cat # 325516. Contains 150 mg Glucose and 150 mg Glutamic acid per liter.

10.10 Nitrification Inhibitor, 2-chlor-6-(trichloromethyl)pyridine: Nitrification inhibitor HACH Formula 2533 (TCMP) or equivalent.

10.11 Sodium Hydroxide, 1N: Dissolve 4 g of sodium hydroxide, NaOH, in 80 mL distilled water. When cool dilute to 100 mL.

10.12 Sulfuric Acid 1 N: Dilute 2.8 mL concentrated sulfuric acid, H_2SO_4, to 100 mL with distilled water. CAUTION: Always add acid to water, NEVER water to acid. This reaction liberates excessive heat and may react violently.

10.13 Sulfuric Acid Solution: Used in determining amount of Sodium Sulfite Solution used in de-chlorination. Dilute 1mL of concentrated H_2SO_4 to 50mL with distilled water. CAUTION: Always add acid to water, NEVER water to acid. This reaction liberates excessive heat and may react violently.

10.14 Distilled Water: Store distilled water as necessary for quality improvement. Store in BOD incubator for at least 12 hours prior to use.

10.15 Dilution Water: Add 1.0 mL magnesium sulfate (8.2), 1.0 mL calcium chloride (8.3), 1.0 mL ferric chloride (8.4) and 1.0 mL phosphate buffer (8.1) for each 1000 mL of distilled water. **Note: The dissolved oxygen level of this water should be between 8.3 - 8.9 mg/L prior to use. If the DO exceeds this range proceed to section 11.4. Document the DO of the dilution water on the Benchsheet.**

10.16 Seed source: Settled domestic waste provided by the Green Bay Metropolitan Sewerage District.

11. Calibration and Standardization

11.1 The dissolved oxygen probe membrane is replaced as needed (usually monthly). However, should bubbles form under the membrane, the membrane must be changed. The probe electrode is cleaned monthly by soaking it in a 15 % v/v ammonium hydroxide solution for 10 minutes.

11.2 The meter is calibrated in water-saturated air following instructions in the operations manual. The dissolved oxygen meter must be calibrated daily (2x/day). Check membrane to see if it needs to be replaced, dry off membrane, and let probe come to equilibrium for 1/2 hour in BOD bottle filled with one inch of distilled water.

11.3 Clean bottles in dishwasher with Lab Solutions Powder detergent (or equivalent) and rinse thoroughly with de-ionized water. Bottles should be completely dry before use.

11.4 Dilution H_2O is initially checked before setting up blanks: one carboy will fill about 1.25 aspirator bottles. These bottles can be filled to a volume that is needed for that day's set-up. One BOD bottle is filled from each aspirator bottle and the DO is determined. If the DO is not with 8.3 to 8.9 mg/L, proceed with one of the following:

11.5 If the DO is < 8.3 mg/L, shake the aspirator bottle until the DO is within 8.3 to 8.9 mg/L.

11.6 If the DO is > 8.9 mg/L, pour 2-3 L of the water to a 4000 mL beaker and heat on a hot plate until DO is reduced. Pour the water back into the aspirator bottle. Pour a portion of the water from the aspirator bottle to a BOD bottle. Read the BOD bottle. If the DO is not within 8.3 to 8.9 mg/L, repeat this step. Use the water when the temperature has returned to 20°C. Alternately, the water can be filtered using Fisher G6 Cat.# 09-804-90A, glass microfiber filters.

11.7 If one of the aspirator bottles has a high DO and one a low DO, mix a portion (4-6 L) together and return the water to the aspirator bottle. Pour a portion of this water into a BOD bottle. Read the BOD bottle, if the DO is within 8.3 to 8.9 mg/L, proceed with the analysis.

11.8 The temperature at which the distilled water is stored should be kept at 20 to 25°C to minimize the above steps.

12. Procedure

12.1 Sample Pre-Treatment

12.1.1 pH Adjustment - Measure and record the pH of the sample in the pH analysis logbook. If the sample does not fall within the range of 6.0 to 8.0, neutralize the sample into this range with sodium hydroxide (8.8), or sulfuric acid (8.9), whichever is appropriate to a pH of 7.0 to 7.2.

12.1.2 Residual Chlorine Removal

12.1.2.1 Testing for Chlorine - Immerse a piece of potassium iodine (KI) starch indicator paper into all samples. A blue color on the indicator paper indicates the presence of chlorine. If residual chlorine is present, the sample must be dechlorinated prior to incubation.

12.1.2.2 Neutralizing Residual Chlorine - For samples in which chlorine residual does not dissipate in a reasonably short time, destroy chlorine residual by adding $NaSO_3$ solution. Determine required volume of $NaSO_3$ solution on a 100mL portion of sample by adding 1 mL (1/50) H_2SO_4 solution, 1mL potassium iodide (KI) solution (10 g/100 mL) per 100mL of sample and titrating with Na_2SO_3 solution to the starch-iodine end point for residual. Add to neutralized sample the proportional volume of $NaSO_3$ solution determined by the above test, mix, and after 10-20 min check sample for residual chlorine. Do not test chlorinated/dechlorinated samples without seeding.

Note: Excess $NaSO_3$ exerts an oxygen demand and reacts slowly with certain organic chloramine compounds that may be present in chlorinated samples.

12.2 Sample Analysis

12.2.1 For Dissolved BOD filter the sample through a glass fiber filter prior to setting up for BOD analysis. A filter blank must be created and analyzed with the filtered BOD sample.

12.2.2 Nitrification for cBOD –Samples to be analyzed for cBOD must include 2 automated shots (0.16 mg) of Nitrification inhibitor (8.7). Add the nitrification inhibitor when the sample bottle is half full of dilution water: this prevents shocking of the seed by the inhibitor.

12.2.3 Sample temperature adjustment – All samples must be brought to 20 ±1°C before making dilutions. Place samples in a water bath set at 20°C for a sufficient amount of time to bring their temperature up to 20°C ± 1°C. Use a calibrated thermometer to verify temperatures.

12.2.4 Sample Dilutions – All samples are prepared for incubation by diluting an appropriate sample volume with dilution water and adding the required amount of seed solution and GGA solution. Samples will produce the most reliable results when the residual DO after incubation is >/= 1 mg/L and the DO uptake is >/= 2 mg/L. Samples should be selected using historical data, if available. If historical data is available, prepare a minimum of 3 dilutions for each sample. If historical data is not available, prepare 4-6 dilutions for each sample. Note: The seed solution must be added after the sample volume is added to the BOD bottle.

12.2.4.1 Seeding

12.2.4.1.1 It is necessary to have a population of microorganisms capable of oxidizing the biodegradable organic matter in a sample.

12.2.4.1.2 The seed is settled domestic wastewater at 20°C up to 6 days old. Store at 4°C when not in use.

12.2.4.1.3 Seed must be added to untreated industrial samples, high temperature samples, disinfected samples, samples with extreme pH values and any samples that were dechlorinated. All other samples need not be seeded: whenever unsure seed the sample.

12.2.4.1.4 After the predetermined sample dilution is added, the seed solution (about 2 to 5 mLs) is added to each BOD bottle. Dilution water must then be added to completely fill the BOD bottle such that insertion of the stopper will displace all air and no air bubbles will remain.

12.2.4.2 Estimate the BOD of the sample and select dilutions that will yield a DO difference of at least 2.0 mg/L and a final DO of at least 1.0 mg/L, using the following as a guideline. (Set up at least three dilutions per sample).

Estimated BOD (mg/L)	Sample Volumes
Less than 10 (river water)	50, 100, 200
10 – 30 (treated effluent)	25, 50, 100
30 – 60	15, 25, 50
60 – 90	10, 15, 25
90 – 150	5, 10, 15
150 – 300 (raw wastewater)	3, 5, 10
300 – 700 (industrial waste)	1, 3, 5
700 – 1500	0.5, 1, 3
1500 – 2500	0.3, 0.5, 1

12.2.4.3 If greater than 150 mL of sample is used, the nutrients must be added directly to the BOD bottle. Add 0.3 mL of each of the following: phosphate buffer, magnesium sulfate, calcium chloride, and ferric chloride.

12.2.4.4 When performing sample dilutions, measure a proper amount of well-mixed sample into a detergent cleaned and distilled water rinsed BOD bottle. Prepare at least 3 dilutions for each sample:

3.0 mL – 25 mL use wide bore pipette
25 mL – 50 mL use a 50.0 mL graduated cylinder
50 mL – 100 mL use a 100 mL graduated cylinder
100 mL – 250 mL use a 250 mL graduated cylinder

Note: Dilutions under 3 mL must be made using serial dilutions.

12.2.4.5 Serial Dilutions

For sample volumes between 0.30 and 3.0 make Dilution A

Dilution A : Pipette 5 mL of sample into a 50 mL graduated cylinder and dilute to 50 mL with dilution water. Pour into a disposable plastic cup labeled Dilution A.

Sample volume needed:

0.30 mL =	Pipette 3.0 mL of Dilution A into a BOD bottle.
0.50 mL =	Pipette 5.0 mL of Dilution A into a BOD bottle.
1.0 mL =	Pipette 10.0 mL of Dilution A into a BOD bottle.
2.0 mL =	Pipette 20.0 mL of Dilution A into a BOD bottle.

For sample volumes below 0.3 mL make Dilution B.

Dilution B: Pipette 5 mL of diluted sample from Dilution A to another 50 mL graduated cylinder and dilute to 50 mL with dilution water. Pour into a disposable plastic cup labeled Dilution B.

Sample volume needed:

0.05 mL =	Pipette 5.0 mL of Dilution B into a BOD bottle.
0.10 mL =	Pipette 10.0 mL of Dilution B into a BOD bottle.
0.20 mL =	Pipette 20.0 mL of Dilution B into a BOD bottle.

12.2.4.6 After pipetting the correct dilution into the BOD bottle, fill the rest of the bottle with dilution water, so the initial DO can be determined.

12.2.5 Soil Samples - weigh out 0.1 – 3 grams of sample depending on sample type. Use historical information when possible. Use dilution water to rinse sample aliquots from the weigh boats into the BOD bottles.

12.2.6 QC Samples – The following QC samples must be included with each sample batch.

12.2.6.1 Sample duplicate – Prepare one sample per batch per matrix (at least 5%) in duplicate to provide a precision check. Duplicate samples are chosen at random (excluding field, equipment, rinse, and trip blanks) unless designated by the client.

12.2.6.2 Dilution water blanks – Prepare 2 BOD bottles of dilution water for each set-up. These blanks will be used to determine the amount of dissolved oxygen in the dilution water, the quality of the dilution water, and the cleanliness of the BOD bottles.

12.2.6.3 Seed Controls – Prepare BOD bottles with 5, 10, 15, and 30 mLs of undiluted seed (from the Green Bay Metropolitan Sewerage District) and dilution water for each set-up. The volume of seed may vary depending on the strength of the seed: use analyst discretion. By determining the BOD of the seed, the seed correction factor can be calculated for each seeded sample. For cBOD batches only, nitrification inhibitor must be added to each of the seed controls. NOTE: Polyseed may be substituted if a supply of treatment plant seed is unavailable.

12.2.6.4 GGA Check (GGA/LCS) – For each batch, prepare 2 BOD bottles with seed solution, 6mL of GGA standard, and fill to capacity.

12.2.7 Determination of initial DO – A calibrated, clean DO Probe is used to determine the initial DO concentration in mg/L. All samples must be free of air bubbles (remove bubbles by taping on the sides of the bottle) prior to inserting the Probe into the BOD bottles. Read the DO directly from the meter when the reading has stabilized. Record the initial DO. Rinse the probe after each set of samples.

12.2.7.1 If the initial DO is < 7.0 mg/L or > 9.3 mg/L at 20°C, shake the BOD bottle containing diluted sample until the DO is within range.

12.2.8 Incubation

12.2.8.1 After the initial DO is recorded, completely fill the BOD bottle with dilution water and insert the stopper so that all of the air is out of the bottle. Place water seal cap on bottle to reduce evaporation.

12.2.8.2 Place all samples and QC checks in the incubator at 20°C ± 1°C for 5 days ± 6 hours. Make sure that each batch of bottles is labeled with the day the final DO is to be determined (5 days).

12.2.9 Determination of final DO

12.2.9.1 A calibrated, clean DO Probe is used to determine the final DO concentration in mg/L. All samples must be free of air bubbles prior to inserting the Probe into the BOD bottles. Read the DO directly from the meter. Record the final DO.

13. Quality Control

13.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 GGA/LCS Check – The GGA check is performed in duplicate. The acceptance limits are 167.5-228.5 mg/L (198 ±30.5 mg/L). If the result is outside of the acceptance limits, flag each sample with "B4" – BOD laboratory control sample not within control limits. Due to the 48 hour holding time for this test, it is not practical to reanalyze and try to correct the deficiency.

13.3 Seed Control – One series of seed controls must be analyzed with every analysis. If performing cBOD, the seed control series must include nitrification inhibitor. The seed control concentration should be 0.6-1.0 mg/L. These limits are for guidance only. All samples that are seeded must be seed corrected.

13.4 Dilution Water Blank Check –

13.4.1 Two dilution water blanks must be tested with each batch.

13.4.2 The DO uptake of a dilution water blank should not be more than 0.2 mg/L and preferably not more than 0.1 mg/L.

13.4.3 If the DO difference is greater than 0.2 mg/L, recalibrate the meter. If the DO difference is still out of range, bleach all of the containers that hold distilled or diluted water. If the DO difference is still out of range, look for other sources of contamination. Note: bleaching these containers should be done upon emptying them and before refilling to prevent this from happening. The carboys are also allowed to air dry before refilling.

13.4.4 If the DO uptake is >0.2 mg/L, all samples and QC must be flagged "B3" – "the dissolved oxygen depletion in the dilution water blank exceeded 0.2 mg/L."

13.5 Duplicates: One sample must be performed in duplicate for each batch of samples and for each matrix in the batch. Duplicate samples are chosen at random (excluding field, equipment, and trip blanks) unless designated by the client. In the absence of project specific requirements, the RPD of duplicates (samples and LCSs) must be </= 20%. If outside this control limit, the parent sample must be flagged with an appropriate "R" qualifier.

13.6 Samples

13.6.1 The initial DO of samples or sample dilutions must be between 7.0-9.4 mg/L. If outside these limits, sample shaken to aerate until in control.

13.6.2 For dilutions that deplete at least 2.0 mg/L and leave at least 1.0 mg/L (final DO) the results are calculated. The average of all acceptable results with at least 2.0 mg/L DO depletion and at least 1.0 mg/L residual DO is reported.

13.6.3 If all dilutions have a final DO reading of <1.0 mg/L, results are reported from the smallest sample dilution. The result is reported as > Estimate and qualified with a "B1" data qualifier – "Less than 1.0 mg/L DO remained for all dilutions set. The reported value is an estimated greater than value and is calculated for the dilution using the least amount of sample." The actual result may be higher.

13.6.4 The reported value is the minimum possible result. The actual result may be higher.

13.6.5 If all of the sample dilutions have DO differences <2.0 mg/L (due to sample depletion), results are reported from the largest sample dilution. The result is reported as < Estimate and qualified with a "B2" data qualifier – "Oxygen usage is less than 2.0 for all dilutions in set. The reported value is an estimated less than value and is calculated for the dilution using the most amount of sample." The actual result may be lower.

13.6.6 Toxic Slides – A sample is considered toxic if the dilutions show higher values in the more dilute concentrations. If toxicity is suspected, report the result with the highest BOD. Flag the result with "Sample exhibited characteristics of a toxic slide."

13.6.7 If the sample is set up after the hold time has expired, the data must be flagged with an appropriate data qualifier.

14. Data Analysis and Calculations

14.1 Determine the BOD result of each sample in mg/L. For soils, the BOD result should be calculated in mg/L soluble.

14.2 Each dilution is calculated out unless the difference between the initial and final reading is < 2.0 mg/L or the final DO is < 1.0 mg/L.

14.2.1 If ALL dilutions have a final DO reading of <1.0 mg/L, the following calculation is used to obtain an ESTIMATE.

14.2.1.1 Use smallest sample volume data.

14.2.1.2 (Initial DO-1.0) – seed correction / Decimal volumetric faction of the sample used = Estimate

14.2.1.3 The result is reported as > EQL (EQL is the determined Estimate) and qualified with a "B1" data qualifier – "Less than 1.0 mg/L DO remained for all dilutions set. The reported value is an estimated greater than value and is calculated for the dilution using the least amount of sample."

14.2.1.4 All dilutions with ≥ 2.0 mg/L DO difference are averaged for the final BOD result except when toxicity is present. If the DO difference between the initial and final reading is <2.0, then that BOD is not used for the average. Note: The individual BOD Concentrations determined using 11.3.5.2 are the values averaged. At least one of the dilutions must have a DO Depletion of ≥2.0 mg/L due to the sample (excluding depletion due to the seed). If not, an estimate is calculated as starting at 11.3.2.3.1.

14.2.1.5 If all dilutions have DO differences < 2.0 mg/L (due to sample depletion and excluding the depletion due to seed), the following calculation is used to obtain an ESTIMATE.

14.2.1.5.1 Use largest sample volume data.

14.2.1.5.2 2.0 x Dilution Factor for dilution at highest volume sample = EQL

14.2.1.5.3 The result is reported as < EQL and qualified with a "B2" data qualifier – "Oxygen usage is less than 2.0 for all dilutions in set. The reported value is an estimated less than value and is calculated for the dilution using the most amount of sample.

14.2.2 The above guidelines are followed as a "general" rule. These guidelines may be invalidated depending on the interpretation of each case. As a general rule, BOD's are not to be reset since the holding time limit of 48 hours would have expired.

14.2.3 When dilution water is not seeded:

$$BOD_5 \text{ mg/L} = D_1 - D_2 / P$$

Where:

D_1 = DO of diluted sample immediately after preparation, mg/L,

D_2 = DO of diluted sample after 5 day incubation at 20°C, mg/L, and

P = decimal volumetric fraction of sample used. The decimal volumetric fraction = sample volume used / total BOD bottle volume (300 mL).

14.2.4 When dilution water is seeded:

14.2.4.1 Determine the Seed Correction (s.c) of the four seed dilutions using the calculation below:

$$\text{s.c.} = \frac{A \times B}{300}$$

Where:

A= the **average** of valid seed control BOD results

B= mLs of seed per 300 mL BOD bottle

14.2.4.2Calculate the final result using the calculation below:

BOD_5 mg/L = $(D_1 – D_2)$ –s.c / P

Where:

D_1 = DO of diluted sample immediately after preparation, mg/L,

D_2 = DO of diluted sample after 5 day incubation at 20°C, mg/L,

P = decimal volumetric fraction of sample used,

s.c. = seed correction (determined in calc 12.3.5.1)

14.2.4.3Calculate the EQL result using the calculation below:

EQL = 2.0 mg/L x Dilution Factor

*The EQL does not need to be corrected for the seed.

14.2.5 Average the BOD mg/L calculated for each dilution to get a BOD mg/L for the sample. Use this value to record the data. When recording data make sure that the proper number of significant digits is used. (See chart below)

If BOD mg/L is:

< 10 mg/L record as X.X
> 10 mg/L record as XX
> 100 mg/L record as XX0
> 1,000 mg/L record as X,X00

14.3 > 10,000 mg/L record as XX,000

15 Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 See Section 13 and Table A.

16 Corrective Actions for Out-of-Control Data

16.1 See Section 13 and Table B.

17 Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 See Section 13 and Table B.

18 Method Performance

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s (most current revision or replacement). A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

18.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of the LOD and LOQ* (most current revision or replacement), for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

18.1.4 Periodic performance evaluation (PE) samples are analyzed per S-ALL-Q-010, *PE/PT Program* (most current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office.

19 Method Modifications

19.1 Not Applicable to this SOP.

20 Instrument/Equipment Maintenance

20.1 Refer to the DO probe manual for any applicable maintenance.

20.2 Document all maintenance in the instrument maintenance logbook.

21 Troubleshooting

21.1 Refer to the DO probe manuals for any applicable troubleshooting.

22 Safety

22.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

22.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Safety Data Sheets (SDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

23 Waste Management

23.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

23.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Handling and Management*.

24 Pollution Prevention

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25 References

25.1 Pace Analytical Quality Assurance Manual (most current revision or replacement).

25.2 Standard Methods for the Examination of Water and Wastewater, on-line ed., Method 5210 B-01, 5-Day BOD Test, 2001, Editorial Revision 2011.

26 Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

26.1 BOD Benchsheet

26.2 Table A: Quality Control

26.3 Table B: Analyst/Technician Data Assessment

27 Revisions

Revision Number	Reason for Change	Date
S-GB-I-044-Rev.0	Updated to Pace Format. Changed Dup QC frequency from 10% to 5%. Changed procedure for making GGA standard. Added BOD and pH bench sheets.	15Feb2007
S-GB-I-044-Rev.01	Updated signature page. Deleted Section 7. Renumbered document. Sections 11 and 12 – Updated data qualifiers Section 13 – Updated SOP references. Section 14 – Updated SOP reference Section 15 – Updated Standard Methods reference	30Jul2008
S-GB-I-044-Rev.02	Updated Signature page Section 9: Updated SOP Reference Section 11.3: Updated all Calculations to match current practices. Changed "Estimate" to EQL for final results. Section 11.3.2.2: Added Note to average BOD values Section 11.3.5.2: Included calculation to determine final BOD value. Section 11.3.5.3: Included calculation to determine EQL. Updated Attachment I: Current BOD Spreadsheet	20Dec2010
S-GB-I-044-Rev.03	Throughout Document: Updated SOP References. Section 11.1.2: Removal of residual chlorine changes made, Section 4.4, 9.7, 11.2.3 and 11.2.8.2: Sample temperature changed from 20±3 C to 20±1C	02Aug2012
S-GB-I-044-Rev.04	Throughout Document: Updated to current SOP format. Sections 6.3, 12.1.1: Changed pH to be within 6.0-8.0, and if not to adjust to 7.0-7.2. Section 10.1.5: Added that the DO of the dilution water must be documented on the Benchsheet. Tables A and B: Added.	03Apr2014

BOD Benchsheet

BOD BENCHSHEET

Method: 5210B-T-1
EQL: 2.0
SLBP/CBP/room temp at set (C): 29.74 740 20.6
SLBP/CBP/room temp at read (C): 29.96 746 20.8
GBM Seed: 1127-39-22
mL GBM seed/Bottle: 3
GBM Seed Correction: #VALUE!
Instrument: YSI 5000 DO Meter S/N 00G31001

Phosphate Buffer: 1127-1-9
Magnesium Sulfate: 1127-83-12
Calcium Chloride: 1127-75-12
Ferric Chloride: 1127-79-12
dil. Water pH: 7.27
Incubator (Circle One): 40INC02/40INC03/40INC04

Set Date/Analyst: 4/30/0 19DEY
Time In: 17:30
Read Date/Analyst: 5/5/09 DEY
Time Out: 12:20
GGA Std #: 1127-37-2

Worksheet Verified:
Worksheet #:

Note: In Seed Column use "S" for GB Metro Seed or "I" for Polyseed
SLBP=bar. pressure at sea level in inches (as reported by Green Bay airport)
CBP=bar. pressure corrected to 594 ft above sea level, expressed in mmHg (SLBP*0.98*25.4)

# of Dil	Seed	Client Name	Sample Date	Prep Time	Pace #	Analytical Test	Matrix	Bottle #	Initial Volume (mL)	Final Volume (mL)	Dil Factor	Std Conc. (mg/L)	Sample Temp (C)	Initial DO	Final DO	DO Differ	Conc. (mg/L)	Mean	% RPD	Std % Rec	Final Conc. (mg/L)	Rep. Result (mg/L)	Sample EQL (mg/L)	Footnote
																		NA	NA					

1. Dilution Water: Add 12.0 mL of each: magnesium sulfate, calcium chloride, ferric chloride and phosphate buffer to a final volume of 12 L in distilled wat

F-GB-I-018-Rev.06 (20Dec2010)

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Table A. QUALITY CONTROL

Analysis Method ⇨ Quality Control Measure ⇩	**SM 5210B**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Laboratory Control Spike and Laboratory Control Spike Duplicate	One pair per batch of samples up to 20 environmental samples.
Duplicate	One per batch of samples, up to 20 environmental samples.
Initial Calibration	

Table B. ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**SM 5210B If these conditions are not achieved ⇒**
Method Blank	• 1
Laboratory Control Spike and Laboratory Control Spike Duplicate	• 2
Duplicate	• 3

1. In the absence of project specific requirements, sample detects less than 20 times the method blank contamination level is reported with the appropriate “B” data qualifier. Sample detects greater than 20 times the method blank contamination are reported without qualification.

2. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and/or precision data qualifier to reflect the deficiencies.

3. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

Pace Analytical Services, Inc.
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STANDARD OPERATING PROCEDURE

Total Phosphorus using Block Digestion and Analyzed by SmartChem

Reference Method: EPA 365.4

SOP NUMBER:	S-GB-I-061-REV.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-061-Rev.02

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 01/20/14

Kate Grams, Laboratory Quality Manager — Date: 1/16/14

Chad Rusch, Department Manager — Date: 1/16/14

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE/IDENTIFICATION OF THE METHOD

This Standard Operating Procedure (SOP) describes the analyses of Total Phosphorus using Block Digestion and Analyzed by SmartChem by EPA 365.4.

2. SUMMARY OF METHOD

2.1 The sample is heated in the presence of sulfuric acid, K_2SO_4 and $HgSO_4$ for two and one half hours. The residue is cooled, diluted to 20 ml and placed on the Auto Analyzer for phosphorus determination. This digestion can also be used for TKN.

2.2 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid which is measured at 880 or 660 nm. The color is proportional to the phosphorus concentration.

3. SCOPE AND APPLICATION

3.1 This SOP is applicable to the analysis of Total Phosphorus in drinking, ground, surface, and saline waters, domestic and industrial wastes, soils, and TCLP, ASTM, and SPLP extracts. The applicable range is MDL to the top standard in the curve. This SOP covers two calibration curves/ranges. The lower of the two ranges is referred to Low Level Phosphorus.

3.2 This SOP utilizes the SmartChem Discrete analyzer.

4. APPLICABLE MATRICES

4.1 Drinking, ground, surface, and saline waters

4.2 Domestic and industrial wastes

4.3 Soils and Solid matrices.

4.4 TCLP, ASTM, and SPLP extracts

5. LIMITS OF DETECTION AND QUANTITATION

5.1 Current MDL and RL can be found in the Laboratory Information Management System (EpicPro) or in the Green Bay Wet Chemistry Method Detection Limit Summary.

5.2 The Low Level Phosphorus calibration curve and standards are a factor of 10 lower than the regular Phosphorus calibration.

6. INTERFERENCES

6.1 Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1 mg/L arsenic may cause a positive error. Treat samples high in arsenates with sodium bisulfite before digestion to eliminate this interference.

6.2 Concentrations of iron greater than 50 mg/L cause a negative error due to competition with the complex for the reducing agent ascorbic acid. Treat samples high in iron with sodium bisulfite before digestion to eliminate this interference, as well as the interference due to arsenates.

6.3 Samples for total phosphorous should only be filtered after digestion. Sample color that absorbs in the photometric range use for analysis will also interfere.

6.4 Glassware washed with commercial detergents can cause a contamination problem. All glassware used should be washed with 1:1 HCl and rinsed with deionized water.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

Table 1: Sample Collection, Preservation and Handling

Matrix	Method	Container(s)	Preservation Hold Time	Shipment Conditions	Lab Storage Conditions
Aqueous	EPA 365.4	250 ml plastic containers	H_2SO_4 / 28 days	On ice ≤6° Celsius	Walk-in cooler ≤6°C
Solid	EPA 365.4	Glass or plastic containers	None / 28 days	On ice ≤6° Celsius	Walk-in cooler ≤6°C

8. DEFINITIONS

8.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

8.2 Reagent Base Line (RBL) – Absorbance created when the diluent and method reagents are added to the cuvette. The diluent is a matrix match of the sample matrix.

8.3 Water Base Line (WBL) – Absorbance created when reagent grade water is added to the cuvette. The WBL is used to check the quality of the cuvette, condition of the filters, and lamp. If the value is too high or too low the cuvette will be rejected and will not be used in the analysis.

9. EQUIPMENT AND SUPPLIES

Table 2: Equipment

Equipment	Manufacturer	**Model(s)/Catalog number***
Westco SmartChem Discrete Analyzer	Westco	Discrete Analyzer
Westco Block Digestor	Westco	40 position
Analytical Balance	Mettler	BA310P
500 µL Pipette	Eppendorf	05-402-76 (Fisher Cat#)
250 µL Pipette	Eppendorf	05-402-72 (Fisher Cat#)
1000 uL Pipette	Eppendorf	05-402-69 (Fisher Cat#)
Repipetors (0.5 - 5.0mL)	Barnstead/Thermolyne	13-687-33 (Fisher Cat#)
Vented Hood	Hamilton	
Spoonula (Lab Spoon)	FisherBrand	14-375-10 (Fisher Cat#)
Glass pipette, graduated, widemouth		
Block Digestor #1	Bran & Lubbe	BD-40
Block Digestor #2	Tecator	2040 Digestor
Volumetric flasks, various		

* Or equivalent

Table 3: SUPPLIES

Supplies	**Vendor**	Catalog number*
Digestion tubes – 75 mL	Bran & Lubbe	
Boiling chips	MG Scientific	B336-1
Culture Tubes 13X100	Fisher	71500 13100
* Or equivalent		

10. REAGENTS AND STANDARDS

Table 4: REAGENTS

Reagent	Alias	Concentration	Directions found in
Ammonium Molybdate Tetrahydrate ($[(NH_4)_6Mo_7O_{24}\bullet 4H_20]$		Neat	
Antimony Potassium Tartrate $[K(SbO)C_4H_4O_6\bullet 1/2H_2O]$		Neat	
Molybdate/Antimony Solution			Table 6
Sulfuric Acid		Concentrated	
Sulfuric Acid Solution		4%	Table 6
Ascorbic Acid		Neat	
Ascorbic Acid Solution			Table 6
Acetone			
Sodium Dodecyl Sulfate		Neat	
Sodium Dodecyl Sulfate Solution	SDS	15%	Table 6
Sodium Chloride		Neat	
Acid / Salt Solution	Reagent A		Table 6
Salt Solution	Reagent B		Table 6
Potassium Sulfate (K_2SO_4)		Neat	
Sulfuric Acid-Mercuric Sulfate-Potassium Sulfate Solution	Digestion Solution		Table 6
Reagent Water	Deionized Water		
Mercuric Oxide Red		Neat	
Mercuric Sulfate Solution			Table 6
Hydrochloric Acid		Concentrated	
Hydrochloric Acid – 6 N	Glassware Rinse Solution	6 N	Table 6
Potassium Hypophosphate (KH_2PO_4)		Neat	
Cuvette Cleaning Solution		Westco Part # 3AS-RN00-20	
Cuvette Cleaning Solution			Table 6
Probe Rinse Solution		Westco Part # 3AS-RN00-21	
Probe Rinse Solution			Table 6
Matrix Solution			Table 6

Table 5: STANDARDS

Standard	Concentration	Direction found in Section:	Alias
Potassium hypophosphate (KH_2PO_4) Dry at 105ºC for 1 hour.	-1000 mg/L -100mg/L(LLPhos)	Table 6	Stock Standard
Potassium hypophosphate (KH_2PO_4) Dry at 105ºC for 1 hour. Must be of different Lot # than Stock.	-1000 mg/L -100mg/L(LLPhos)	Table 6	Second Source Stock Standard
Level 1 Calibration Standard	20 mg/L	Table 6	Cal Std. 1
Level 2 Calibration Standard	10 mg/L	Diluted by Instrument from Cal Std. 1	Cal Std. 2
Level 3 Calibration Standard	5.0 mg/L	Diluted by Instrument from Cal Std. 1	Cal Std. 3
Level 4 Calibration Standard	2.0 mg/L	Diluted by Instrument from Cal Std. 1	Cal Std. 4
Level 5 Calibration Standard	1.0 mg/L	Diluted by Instrument from Cal Std. 1	Cal Std. 5
Level 6 Calibration Standard and RLVS standard	0.40 mg/L	Diluted by Instrument from Cal Std. 1	Cal Std. 6
Level 7 Calibration Standard	0.0 mg/L		Cal Std. 7
Initial Check Verification	5.0 mg/L	Table 6	ICV
Continuing Check Verification	10 mg/L	Table 6	CCV
Laboratory Control Spike/Laboratory Control Spike Duplicate	5.0 mg/L	Table 6	LCS/LCSD
Matrix Spike/Matrix Spike Duplicate	5.0 mg/L	Table 6	MS/MSD
Low Level, Level 1 Calibration Standard	2.0 mg/L	Table 6	LL Cal Std. 1
Low Level, Level 2 Calibration Standard	1.0 mg/L	Diluted by Instrument from LL Cal Std. 1	LL Cal Std. 2
Low Level, Level 3 Calibration Standard	0.50 mg/L	Diluted by Instrument from LL Cal Std. 1	LL Cal Std. 3
Low Level, Level 4 Calibration Standard	0.20 mg/L	Diluted by Instrument from LL Cal Std. 1	LL Cal Std. 4
Low Level, Level 5 Calibration Standard	0.10 mg/L	Diluted by Instrument from LL Cal Std. 1	LL Cal Std. 5
Low Level, Level 6 Calibration Standard and RLVS standard	0.04 mg/L	Diluted by Instrument from LL Cal Std. 1	LL Cal Std. 6
Low Level, Level 7 Calibration Standard	0.0 mg/L		LL Cal Std. 7
Low Level Initial Check Verification	0.5 mg/L	Table 6	LL ICV
Low Level Continuing Check Verification	1.0 mg/L	Table 6	LL CCV
Low Level Laboratory Control Spike/Laboratory Control Spike Duplicate	.5 mg/L	Table 6	LL LCS/LCSD
Low Level Matrix Spike/Matrix Spike Duplicate	0.5 mg/L	Table 6	LL MS/MSD

Table 6: PREPARATION OF STANDARDS

Standard	Alias	Final Concentration	Directions	Final Volume
Potassium hypophosphate (KH_2PO_4)	Stock Standard	-1000 mg/L -100mg/L(LLPhos)	Dissolve 4.394 g (0.4394g for LLPhos) of potassium hypophosphate KH_2PO_4 dried at 105˚C for 1 hour in 900 ml of reagent water. Add 2 ml of $conc_c.H_2SO_4$ and dilute to 1 $liter_o$	1000 ml
Level 1 Calibration Standard	Cal Std. 1	20 mg/L	Pipette 2.0 ml of stock standard to 100 ml volumetric flask. Dilute to volume with matrix solution. Prepare fresh monthly.	100 ml
Level 1 Low Level Calibration Standard	LL Cal Std. 1	2 mg/L	Pipette 0.2 ml of stock standard to 100 ml volumetric flask. Dilute to volume with matrix solution. Prepare fresh monthly.	100 ml
Initial Check Verification	ICV	5.0 mg/L	Pipette 0.5 ml of second source stock standard to 100 ml volumetric flask. Dilute to volume with matrix solution. Prepare fresh daily.	100 ml
Low Level Initial Check Verification	LL ICV	0.5 mg/L	Pipette 0.05 ml of second source stock standard to 100 ml volumetric flask. Dilute to volume with matrix solution. Prepare fresh daily.	100 ml
Continuing Check Verification	CCV	10 mg/L	Pipette 1.0 ml of stock standard to 100 ml volumetric flask. Dilute to volume with matrix solution. Prepare fresh daily.	100 ml
Low Level Continuing Check Verification	LL CCV	1.0 mg/L	Pipette 0.1 ml of stock standard to 100 ml volumetric flask. Dilute to volume with matrix solution. Prepare fresh daily.	100 ml
Laboratory Control Spike/ Laboratory Control Spike Duplicate	LCS/LCSD	5.0 mg/L	Pipette 0.1 ml of second source stock standard into digestion tubes. Add 20 ml of DI water and 5 ml of digestion solution. After digestion dilute to 20 ml volume with DI water.	20 ml
Low Level Laboratory Control Spike/ Laboratory Control Spike Duplicate	LL LCS/LCSD	0.5 mg/L	Pipette 0.01 ml of second source stock standard into digestion tubes. Add 20 ml of DI water and 5 ml of digestion solution. After digestion dilute to 20 ml volume with DI water.	20 ml
Matrix Spike/ Matrix Spike Duplicate	MS/MSD	5.0 mg/L	Pipette 0.1 ml of second source stock standard into digestion tubes. Add 20 ml DI water and 5 ml of digestion solution. After digestion dilute to 20 ml volume with DI water.	20 ml
Low Level Matrix Spike/ Matrix Spike Duplicate	LL MS/MSD	0.5 mg/L	Pipette 0.01 ml of second source stock standard into digestion tubes. Add 20 ml DI water and 5 ml of digestion solution. After digestion dilute to 20 ml volume with DI water.	20 ml

Table 7: PREPARATION OF REAGENTS

Reagent	Alias	Concentration	Directions	Final Volume
Sodium Dodecyl Sulfate Solution		15% w/w	Dissolve 15 g of SDS $CH_3(CH_2)_{11}OSO_{33}Na$ in 85 ml of reagent water. Stirring and heating may be required to completely dissolve the SDS.	85 ml
Molybdate/Antimony Solution			Dissolve 2 g of ammonium molybdate tetrahydrate, $(NH_4)_6Mo_7O_{24}4H_2O$ and 0.084 g of antimony potassium tartrate $K(SbO)C_4H_4O_6$ $1/2H_2O$ in about 200 ml reagent water. Add 10 ml of 15% w/w SDS and dilute to 250 ml with reagent water. Invert five times to mix. Store solution in an acid washed plastic bottle at room temperature.	250 ml
Sulfuric Acid Solution		4% v/v	Slowly add 40 ml of conc. sulfuric acid to 800 ml of reagent water, cool and dilute to 1 liter. Use this solution as the RB (diluents) if a digested blank is not available. The digested blank from either digestion procedure should be used as the RBL and diluents for the SmartChem auto-preparation	1000 ml
Ascorbic Acid Solution			Dissolve 3 g of ascorbic acid $C_6H_8O_6$ in about 30 ml of reagent water. Add 0.1 ml of acetone CH_3COCH_3 and 0.5 ml of 15% w/w SDS dilute to 50 ml with reagent water. Invert five times to mix. Store solution in an acid washed plastic bottle at room temperature.	50 ml
Mercuric Sulfate Solution			Dissolve 8 g red mercuric oxide HgO in 50 ml of 1:4 sulfuric acid and then dilute to 100 ml. To prepare the 1:4 sulfuric acid solution, carefully add 10 ml conc. H_2SO_4 to 40 ml reagent water.	100 ml
Digestion Solution	Sulfuric acid-mercuric sulfate-potassium sulfate solution		Dissolve 133 g K_2SO_4 in 700 ml of reagent water and then slowly add 200 ml conc. H_2SO_4. Allow the solution to cool and then add 25 ml of mercuric sulfate solution and dilute to 1 liter	1000 ml
Acid / Salt Solution	Reagent A		Dissolve 2.65 g of NaCl in 450 ml of reagent water in a 500 ml volumetric flask. Slowly add 9.0 ml of conc. sulfuric acid while swirling the flask. Mix and allow the solution to cool to room temperature and then add 10 ml of 15% w/w SDS and dilute to 500 ml with reagent water. Invert five times to mix. Store Reagent A in an acid washed plastic bottle at room temperature.	500 ml
Salt Solution	Reagent B		Dissolve 2.65 g of sodium chloride NaCl in 450 ml of reagent water and then add 10 ml of 15% w/w SDS and dilute to 500 ml with reagent water. Invert five times to mix. Store Reagent B in an acid washed plastic bottle at room temperature.	500 ml
Hydrochloric Acid	Glassware Rinse Solution	6 N	Add 500 ml concentrated hydrochloric acid into ~ 400 ml DI water. Dilute to volume.	1000 ml
Cuvette Cleaning Solution			Add 50 ml of cuvette cleaning solution to approximately 700 ml of reagent water and dilute to 1 liter with reagent water. Invert five times to mix. Store the solution at room temperature.	1000 ml
Probe Rinse Solution			Add 0.5 ml of probe rinse solution and dilute to 1 liter of reagent water. Invert five times to mix. Store the solution at room temperature.	1000 ml
Matrix Solution			Add 240 ml digestion solution to 1 liter volumetric flask. Dilute to volume with DI water.	1000 ml

11. CALIBRATION

11.1 **Daily Calibration** – The SmartChem must be calibrated each time it is set up and at least every 24 hours that samples are analyzed. A linear calibration is used. Calibration consists of a minimum of 5 standards plus a blank. The working range is from the current MDL to the highest standard in the calibration curve.

11.2 **Calibration Verification** – Each calibration must be verified by analyzing an Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB). A Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) must also be analyzed after each set of 10 samples.

11.3 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the Pace Reporting Limit. The CRDL is used to show analyte recovery at the reporting limit.

11.4 **Acceptance Criteria** – The correlation coefficient of the response for elements requiring multiple levels must be ≥ 0.995. The results of the ICV/CCV checks must agree within ± 10%. The results of the ICB/CCBs must be <RL.

11.5 Follow steps in section 12 to run calibration curve.

11.6 The SmartChem can automatically prepare diluted standards and samples, as required, to bring high concentration standards and samples into the working range of the method. See the SmartChem Operation Manual Chapter 3 for instructions on how to configure the SmartChem Parameter Method File.

12. PROCEDURE

NOTE: Document all sample volumes, standards and reagents used in the digestion on the prep log.
NOTE: Rinse all glassware with a 50% solution of HCl followed by DI water rinse before each use.

12.1 **Digestion Procedure** – This portion of the SOP describes the steps to digest the samples before analysis. This digestion procedure is the same digestion procedure used for total kjedahl nitrogen analysis. The digestate can be used for both total kjedahl nitrogen and total phosphorus analysis without individual digestions.

12.1.1 Add 3-4 Teflon boiling chips to a digestion tube. (Too many boiling chips may cause the sample to boil over.)

12.1.2 Aqueous – Pipette, using clean wide-mouth graduated glass pipette, 20 ml of sample into the digestion tube.

12.1.3 Solid – Weigh out 0.20 g – 0.50g of sample to the nearest 100th on a calibrated balance and add to digestion tube. The sample weight is dependent upon the sample matrix. Bring to volume of about 20 ml with DI water. Mix the sample with a vortex mixer

12.1.4 For liquid samples, prepare one method blank by adding 20 ml of DI water to digestion tube using a calibrated automatic pipette.

12.1.5 For soil samples, prepare one method blank by weighing out 0.20 g of Teflon chips to the nearest 100th on a calibrated balance and add to digestion tube. Bring to volume of about 20 ml with DI water.

12.1.6 For liquid samples, prepare one LCS (and LCSD if needed) by adding 20 ml DI water and 0.1 ml (0.01 mL for LL) of 1000 mg/L second source stock standard into digestion tube using calibrated automatic pipette. A LCSD is only necessary when requested by client. See Table 6.

12.1.7 For soil samples, prepare one LCS (and LCSD if needed) by weighing out 0.20 g Teflon chips to the nearest 100th on a calibrated balance and add to digestion tube. Add 20 ml DI water and 0.1 ml (0.01 mL for LL) of 1000 mg/L second source stock standard using calibrated automatic pipette. A LCSD is only necessary when requested by client. See Table 6.

12.1.8 For liquid samples, prepare one MS/MSD pair per 10 samples by adding 20 ml sample and 0.1 ml of 1000 mg/L second source stock standard into digestion tube using calibrated automatic pipette. See Table 6.

12.1.9 For soil samples, prepare one MS/MSD pair per 10 samples by weighing out 0.20 g of sample to the nearest 100th on a calibrated balance and add to digestion tube. Add 20 ml DI water and 0.1 ml (0.01 mL for LL) of 1000 mg/L second source stock standard using calibrated automatic pipette. See Table 6.

12.1.10 Using repipettor, add 5 ml mercuric sulfate/potassium sulfate digestion solution. Vortex samples to mix.

12.1.11 Place tubes in block digestor and press run on control panel. The digestor will heat to 160˚C and maintain temperature for 1 hour. The temperature will reset to 380˚C and continue to heat for 1½ hours.

12.1.12 Remove digestion tubes, cool and dilute the liquid residue back up to 20 ml with DI water. Vortex and pour into cuvette. If samples have large particulate matter, filter prior to analysis. Store in refrigerator until analysis. Analyze within 5 days.

12.1.13 Prepare calibration curve and check standards as described in Tables 5 and 6 of section 10.

12.2 **Method Parameters** – The method is equivalent to the EPA protocol 365.4. Modifications to this test method may void EPA equivalency. If modifications are made, then the analyst must perform the required testing to document the method performance in accordance with USEPA requirements. See Table 10 Method Parameters

12.3 **Basic System Operation (Analytical)** - This portion of the SOP is designed to allow the user to set up and run a method. For a more detailed explanation of the many other options, the user should refer to the SmartChem Software Reference Manual. It is assumed that a method program has already been created for the chemistries you wish to run.

12.3.1 If not already on, turn on computer, printer, monitor, and the SmartChem instrument. The SmartChem on/off toggle switch is located on the left side of the instrument.

12.3.2 Log into the PC and Network. Click twice on SmartChem New and enter the username: *SmartChem New WESTCO* and password: *JOE*.

12.3.3 Build Sequence in LIMSLink of batched samples:

12.3.3.1 Go into LIMSLink and click on the "*Running Man*".

12.3.3.2 Select the *Prep* method and click *Start New* followed by *OK*.

12.3.3.3 In the first two lines enter ICVI followed ICB in the third line and CRDL in the fourth.

12.3.3.4 Click on Get Samples and enter the Batch, the Instrument ID (40WTA9) and click OK.

12.3.3.5 Repeat the last step for each batch of samples in the sequence.

12.3.3.6 Export to SmartChem – click on Import Instrument, Export to SmartChem

12.3.4 SmartChem Sequence Loading and Analysis:

12.3.4.1 Go to Sample Entry.

12.3.4.2 Double click on the desired Method to be run. For Phos/Phos.LL the method is ***P101912-EPA365.4-40WTA9-DAW***.

12.3.4.3 Click on Import from file button

12.3.4.4 Load Sequence file (create Run Plan) - open the SmartChem folder, scroll all the way to the right, and double click on *SmartChem*.

12.3.4.5 Save – Click on the red disk to save with the format ***P101912-EPA365.4-40WTA9-DAW***. 073112 is the month, day, year of the analysis. 40WTA9 is the instrument. WETA is the QUEUE and CCR is an example of analyst initials. Spaces must be used where they are. Click OK.

12.3.4.6 Save External File by clicking *YES* and Save the run plan as ***P101912***.

12.3.4.7 Go to System Monitor.

12.3.4.8 Select and Click on the Run Plan that was created.

12.3.4.9 Verify that the selected Run Plan is correct.

12.3.4.10 Load Samples, Standards, Controls, Diluent, and Empty Cups as displayed in the System Monitor.

12.3.4.11 Check the Probe Rinse, DI Water and Cleaning Solution bottles.

12.3.4.12 NOTE: When filling the Probe Rinse and Cuvette Wash Reservoirs, pour the solutions slowly to minimize foaming. If the probe, DI Water, and/or Cuvette Wash reservoirs require filling while a run is in progress, do not remove the cap and lift the siphon tube above the liquid surface. Insert a long stem funnel through the open port in the reservoir cap and fill the container slowly with the respective solution to minimize foaming.

12.3.4.13 Click Start in System Monitor to display user selectable options before beginning the analysis.

12.3.4.14 If a Wash Cuvette and/or WBL is required before the start of the analysis, then click on the appropriate button. It is recommended that a Wash Cuvette and WBL be run at the start of each day.

12.3.4.15 NOTE: It is recommended that a new WBL be run after a Wash Cuvette operation is performed and/or after the removal and/or replacement of the same or a new cuvette into the reaction tray.

12.3.4.16 Click the Start Button to begin analysis. A box will open. Make sure the Calibration and RBL options are selected for a run that is using a new calibration and click OK.

12.4 **Adding new batches on a Sequence:** Add an additional letter to the name when saving. This is so it will not overwrite the original file. Deselect the *Calibration* and *RBL* boxes if the calibration is not going to be re-analyzed.

12.5 **Shutdown:**

12.5.1 Click on the EXIT icon.

12.5.2 Turn off computer, printer and switch on surge suppresser.

13 QUALITY CONTROL

13.1 Refer to the most current version of the Pace Quality Manual Attachment I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 **Initial Calibration Verification (ICV)**

13.2.1 The ICV must be analyzed before samples.

13.2.2 Concentration must be within $\pm$ 10% of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified.

13.2.3 If the ICV is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.2.4 The lot number of the Potassium hypophosphate Standard used to make the ICV must be different from that of the calibration curve standards.

13.3 **Continuing Calibration Verification (CCV)**

13.3.1 The CCV is analyzed after every 10 samples.

13.3.2 Concentration must be within ± 10% of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified. If the reset CCV fails, recalibrate and reanalyze all samples back to the last acceptable CCV.

13.3.3 If the CCV is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.3.4 The lot number of the Potassium hypophosphate Standard used to make the CCV must be different from that of the ICV.

13.4 **Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)**

13.4.1 The ICB must be analyzed after the ICV and before samples. The CCB must be analyzed after the CCV and before samples.

13.4.2 The absolute value must be < PRL. When measurements are above the PRL, terminate analysis; correct the problem, and the calibration re-verified. If the reset of the ICB/CCB fails, recalibrate and reanalyze all analytical samples analyzed since the last compliant calibration blank.

13.4.3 If the sample concentration is greater than ten times the concentration in the ICB or CCB, the samples do not need to be reanalyzed.

13.5 **Reporting Limit Verification Standard (RLVS)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.6 **Method Blank (MB)**

13.6.1 A MB is carried through all prep procedures and analyzed with a frequency of 5% or one per batch of up to 20 environmental samples. One MB must be digested with each batch.

13.6.2 The absolute value must be < PRL.

13.6.2.1 When measurements are above the PRL, terminate analysis, correct the problem, verify the calibration, and reanalyze all analytical samples analyzed since the last compliant calibration blank.

13.6.2.2 If the analyte is detected in the method blank, method blank criteria is evaluated and flagged with appropriate qualifier to the MDL when MDL reporting is required: qualify with appropriate qualifier sample results less than 10 times the absolute value detected in the blank. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample-by-sample basis. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

13.7 **Laboratory Control Sample (LCS)**

13.7.1 The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples. A Laboratory Control Spike Duplicate (LCSD) must be analyzed if there is insufficient sample volume to perform a matrix spike/matrix spike duplicate or if the client requests one. Must be digested with batch.

13.7.2 Concentration must be within ± 10% of the true value for waters. For soils, the concentration must be within calculated in –house limits or default limits of + 20%.

Accuracy Calculation: $\text{LCS \% Recovery} = \frac{(\text{LCS})}{\text{SA}} * 100$

LCS = LCS Result
SA = Spike Added

Precision Calculation: See section 13.8

13.7.2.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.2.2 If no errors are found and sufficient sample is available, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still out side the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.7.2.3 If sufficient sample volume is not available, report the sample data with appropriate qualifier on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.7.3 When an LCSD is performed, the precision between the LCS and LCSD must be =/< 20% RPD.

13.7.3.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.3.2 If no calculation errors are found when measurements are outside the control limits, flag the associated samples with appropriate data qualifier.

13.7.3.3 The lot number of the Potassium hypophosphate Standard used to make the CCV must be different from that of the LCS.

13.8 **Matrix Spike (MS) and Matrix Spike Duplicate (MSD)**

13.8.1 One pair per up to 10 environmental samples or 10% frequency whichever is more frequent. Must be digested with batch.

13.8.2 Both QC samples must be calculated for accuracy and precision.

Accuracy Calculation: $$\text{Spike Percent Recovery} = \frac{(SSR - SR)}{SA} * 100$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

The relative percent difference (RPD) is calculated as: $$\textbf{RPD} = \frac{|S - D|}{(S + D)/2} * 100$$

where **S** = sample value, mg/L
D = duplicate value, mg/L

13.8.3 Concentration must be within $\pm$ 10% of the true value for waters. For soils, the concentration must be within calculated in –house limits or default limits of $\pm$ 20%.

13.8.4 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.5 If the four times the concentration of the spike is less than the level of the parent, accuracy need not be calculated.

13.8.6 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate qualifier.

13.8.7 The precision between the MS and MSD must be =/< 20% RPD.

13.8.7.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.7.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate qualifier.

13.8.8 The parent sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD.

13.8.9 The lot number of the Potassium hypophosphate Standard used to make the CCV must be different from that of the MS/MSD.

13.9 The appropriate leach blank must be digested and analyzed along with the appropriate samples as leached on the same leach date. The absolute value must be < PRL. When measurements are above the PRL, terminate analysis, investigate and correct the problem, verify the calibration, and reanalyze all analytical samples analyzed.

13.10 When preparation of a sample exceeds 28 days past the time of collection, notify the project manager before proceeding. If a sample is run past 28 days after collection, flag with appropriate data qualifier.

13.11 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with appropriate data qualifier.

14 DATA ANALYSIS AND CALCULATIONS

14.1 See Section 13 for additional calculations. The instrument provides calculated sample results in mg/L. Calculations are only necessary if a dilution was used

Liquid Calculation

Raw Data Value (mg/L) x Dilution Factor = Phosphorous (mg/L)

Soil Calculation

$$\frac{\text{Raw Data Value (mg/L)} * 0.020\ \text{L} * \text{Dilution Factor}}{(0.0002\ \text{Kg} * \%\ \text{solids in decimal form})} = \text{Phosphorus (mg/Kg)}$$

14.2 SmartChem SW will construct and display the calibration curve and calculate all sample results. When the SmartChem is programmed to auto-rerun over-ranged samples, or perform a sample pre-dilution, the dilution corrected results will be reported based on the dilution factors used in the results section of the report. The calibration curve can be reconstructed by plotting instrument response (optical density or absorbance) against standard concentration. Compute sample concentration by comparing the sample response with the standard curve. Multiply calculated sample concentrations by appropriate dilution factor used.

14.3 The concentration value of a sample reported for an over-ranged sample by SmartChem before it is re-run at a pre-selected dilution factor in the Method File is an “estimate” and should not be considered an accurate value since the sample exceeded the highest standard concentration used to generate the calibration curve equation.

14.4 The concentration value of a sample reported for an over-ranged sample by SmartChem before it is re-run at a pre-selected dilution factor in the Method File is an “estimate” and should not be considered an accurate value since the sample exceeded the highest standard concentration used to generate the calibration curve equation.

14.5 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed. Results lower than the MDL should be reported as required by your laboratory certification agency.

15 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

15.1 See Section 13: Quality Control and Table 8: QC Summary

16 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1 See Table 9 Corrective Action

17 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 See Table 9 Corrective Action and Attachment III: Flow Chart

18 METHOD PERFORMANCE

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

18.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of LOD and LOQ*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

18.1.4 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

18.1.5 Linear Calibration Range (LCR): The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to ensure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by +/- 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion

18.1.5.1 Criteria: All standards must be within +/- 10% of the true value with the exception of the highest standard analyzed over the calibration range that should be outside the 10% rule.

18.1.6 Pace Analytical Services, Inc – Green Bay Laboratory will not use any data over the highest calibration standard used. All samples will be diluted and reanalyzed that are over the calibration range.

19 METHOD MODIFICATIONS

19.1 Modifications should be targeted to improve quality, effi*ciency or the* cost effectiveness of the procedure.

19.2 All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.

19.3 Procedures identified as "Best Practices" by PACE 3P Programs will be incorporated into this document as minimum requirements for Pace Laboratories.

19.4 Method EPA 365.4 has been modified to include a digestion procedure for soil and solid matrices.

19.5 If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the "M5" data qualifier.

20 INSTRUMENT/EQUIPMENT MAINTENANCE

20.1 Any daily or periodic maintenance must be recorded in the instrument daily logbook. Additional information may be found in the most current revision of SOP: S-GB-Q-008, *Preventative, Routine, and Non-routine Maintenance.*

21 TROUBLESHOOTING

21.1 Please see the instrument manual for information on instrument troubleshooting.

22 SAFETY

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23 WASTE MANAGEMENT

23.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24 POLLUTION PREVENTION

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25 REFERENCES

25.1 EPA 600 4-79-020, Revised March 1983, Method 365.4

25.2 Standard Methods for the Examination of Water and Wastewater, Phosphorous – SM 4500-P A, B, and F -2011)

25.3 SmartChem Method #411N-0405C

26 TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

26.1 Table 8: **QUALITY CONTOL**

26.2 Table 9: **ANALYST/TECHNICIAN DATA ASSESSMENT**

26.3 Table 10: **METHOD PARAMETERS**

26.4 Flowchart

27 REVISIONS

Revision Number	Reason for Change	Date
S-GB-I-061-Rev.0	First Issue.	11Jan2010
S-GB-I-061-Rev.01	Section 5.3 and 14.3: Updated SOP Number of Waste Handling and Management SOP to S-GB-S-006 Section 11.4: Removed use of Quadratic Curve Fit.	13Sept2010
S-GB-I-061-Rev.02	Updated SOP references throughout document. Throughout Document: Updated SOP format to be consistent with SOP: S-GB-Q-017 *Preparation of SOPs.* Throughout Document: Incorporated Low Level analysis procedure. Throughout Document: Updated Tables to have continuous numbering system, then updated all sections which referenced tables to correct one. Section 12: Updated procedure to SmartChem (from Lachat) operating instructions. Section 25.2: Updated SM Reference to 2011 Edition Section 26: Removed TPO4 Spreadsheet, added Table 10.	23Oct2012
S-GB-I-061-Rev.03	Section 12.1.3: Clarified sample volume used. Section 18.1.5: Updated LDR study verification to every 6 months. Section 19: Additional language to method modifications included for Pace 3P and MS/MSD use..	16Jan2014

Table 8: QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**EPA 365.4**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent. Must be digested with batch.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. A LCSD is required if MS/MSD is not performed. Must be digested with batch.
Matrix Spike / Matrix Spike Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent. Must be digested with batch.
Initial Calibration	Minimum of 5 standards plus blank. Must be performed every time before samples are analyzed.
RLVS Standard	After the calibration
Calibration Verification (ICV/CCV)	ICV – analyzed after calibration but before samples. CCV – analyzed after every 10 samples.
Calibration Blank (ICB/CCB)	ICB – analyzed after ICV. CCB – analyzed after every CCV pair.
Leach Blank	The appropriate leach blank must be digested and analyzed along with the appropriate samples as leached on the same leach date.

Table 9: ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**EPA 365.4 If these conditions are not achieved ⇒**
Method Blank and Leach Blanks	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
RLVS standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 times the method blank contamination are reported without qualification.

2. In the absence of project specific or method requirements, in-house generated limits will be used. If the concentration of the spike is less than 25% of the concentration of the parent, dilute parent to a level that the subsequent spike concentration will be greater than 25% the parent and so the parent concentration is above the EQL. Rerun the parent, MS, and MSD at this new dilution. If the MS and/or MSD fails at the diluted concentration, appropriately flag the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, appropriately flag the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifier to reflect the deficiencies.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB the samples do not have to be reanalyzed.

FLOWCHART

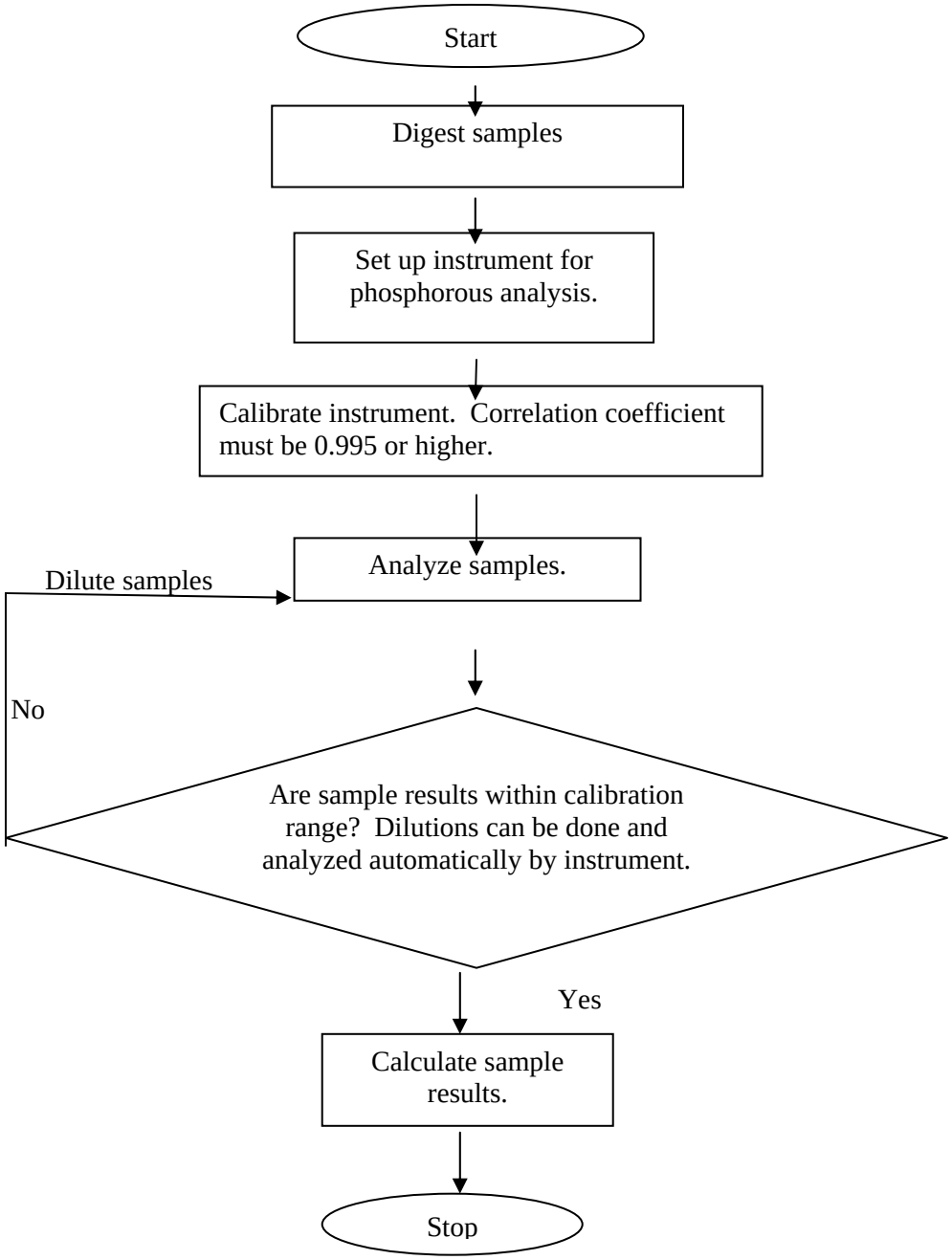
Start
Digest samples
Set up instrument for phosphorous analysis.
Calibrate instrument. Correlation coefficient must be 0.995 or higher.
Analyze samples.
Dilute samples
No
Are sample results within calibration range? Dilutions can be done and analyzed automatically by instrument.
Yes
Calculate sample results.
Stop

Table 10
Method Parameters

Type	End Point
Direction	Direct
Decimals	3
Model	Linear
Filter 1	880 or 660 nm
Sample Blanking	No or "Yes after Reagent 1
Calibration Codes	OP4H and OP4L

High Range TP Method 0.05 – 20.00 mg/L P Hg digestion					
Method Code: WTPH **Range: 0.05 – 20.00 mg/L P**	**Volume** **µL**	**Delay Time** **sec.**	**Read Time** **sec.**	**Rinse** **µL**	**Code**
Sample Volume	40				
Reagent 1-**Acid / Salt Reagent**	264	36	0	0	TPRA
Reagent 2 – Molybdate	64	0	0	0	TPMO
Reagent 3 – Ascorbic Acid	106	0	306	0	TPAS
Maximum Total Reaction	670				
Minimum Sample and	3				

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STANDARD OPERATING PROCEDURE

Total Cyanide using Micro-Distillation and SmartChem

Reference Method: SW846 9012B and EPA 335.4

SOP NUMBER:	S-GB-I-064-REV.02
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-064-Rev.01

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 01/20/14

Kate Grams, Laboratory Quality Manager — Date: 1/16/14

Chad Rusch, Department Manager — Date: 1/16/14

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose/Identification of the Method

This Standard Operating Procedure (SOP) describes the analyses of Total Cyanide using Micro-Distillation and Analyzed by SmartChem Discrete analyzer using methods SW846 9012B and EPA Method 335.4.

2. Summary of Method

2.1 By means of a passive miniature distillation device, MICRO DIST, cyanide in the sample is released by digesting and acidifying cyanide complexes, and converting them to hydrocyanic acid (HCN). The cyanide ion is trapped in a 1.0 M sodium hydroxide absorbing solution, which is diluted to 0.25M solution during distillation. By means of discrete analysis, the 0.25M Noah distillate is converted to cyanogens chloride by reaction with chloramines-T, pyridine and barbituric acid to give a red-colored complex. The absorbance of this complex is measured at 570 nm by measuring the peak area resulting from the sample. The peak area is proportional to the concentration of the cyanide in the sample.

2.2 All samples must be distilled before analysis.

3. Scope and Application

3.1 Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical and distillation methods.

3.2 Parameters: This SOP is applicable to the analysis of Cyanide in drinking, ground, surface, and saline waters, domestic and industrial wastes, soils, and TCLP, SPLP, ASTM extracts.

3.3 This SOP utilizes the SmartChem Discrete analyzer.

4. Applicable Matrices

4.1 Drinking, ground, surface, and saline waters

4.2 Domestic and industrial wastes, soils.

4.3 TCLP, SPLP, ASTM extracts

5. Limits of Detection and Quantitation

5.1 For aqueous samples the current Pace Reporting Limit is 0.02 mg/L. The current MDL is listed in the LIMs and is available by request from the Quality Department.

5.2 For solid samples the current Pace Reporting Limit is 0.6 mg/Kg. The current MDL is listed in the LIMs and is available by request from the Quality Department.

6. Interferences

6.1 Most non-volatile interferences are eliminated or minimized by the distillation procedure. Some of the known interferences are aldehyde, nitrate-nitrite, and oxidizing agents, such as chlorine, thiosulfate, and sulfide.

6.2 Oxidizing agents such as chlorine decompose most cyanide. Test a drop of the sample with potassium iodine-starch paper (KI-starch paper) at time of collection; a blue color indicates need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color changes; then add an additional 0.06 g ascorbic acid per liter of sample.

6.3 False positive results may be obtained by samples that contain Nitrate/or nitrite. Sulfamic acid is added to all samples prior to distillation to eliminate this interference.

6.4 Sulfides adversely affect the procedure by producing hydrogen sulfide during distillation. If a drop of sample on lead acetate paper indicated the presence of sulfide, treat the stabilized sample (pH >12) with bismuth nitrate. Repeat until a drop on the lead acetate paper does not darken. Filter sample to remove precipitate and use filtrate as sample to be distilled.

7. Sample Collection, Preservation, Shipment and Storage

Table 1: Sample Collection, Preservation and Handling

Matrix	Prep Method	Container(s)	Preservation Hold Time	Shipment Conditions	Lab Storage Conditions
Aqueous	SW-846 9012B EPA 335.4	250 ml plastic containers	pH >12; NaOH 14 days	On ice 4+/- 2°C	≤6°C
TCLP, SPLP, ASTM	SW-846 9012B EPA 335.4	glass or plastic containers (when solid) Leachate is filtered into a 250 ml plastic container.	pH >12; NaOH 14 days to extract, additional 14 days to analyze (Total 28 days from collection)	On ice 4+/- 2°C	≤6°C
Solid	SW-846 9012B	glass or plastic containers	None 14 days	On ice 4+/- 2°C	≤6°C

8. Definitions

8.1 Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

8.2 Reagent Base Line (RBL) – Absorbance created when the diluent and method reagents are added to the cuvette. The diluent is a matrix match of the sample matrix.

8.3 Water Base Line (WBL) – Absorbance created when reagent grade water is added to the cuvette. The WBL is used to check the quality of the cuvette, condition of the filters, and lamp. If the value is too high or too low the cuvette will be rejected and will not be used in the analysis.

9. Equipment and Supplies

Table 2: EQUIPMENT

Equipment	Manufacturer	Model(s)/Catalog number •
SmartChem	SmartChem	SmartChem
Micro-Distillation	Lachat	Micro Distillation
Top loading Analytical Balance	Mettler	BA310P
1000 µL Pipette	Eppendorf	05-402-76 (Fisher Cat#)
500 µL Pipette	Eppendorf	05-402-72 (Fisher Cat#)
250 µL Pipette	Eppendorf	05-402-69 (Fisher Cat#)
Repipetors (0.5 - 5.0mL)	Barnstead/Thermolyne	13-687-33 (Fisher Cat#)
Vented Hood	Hamilton	
Spoonula (Lab Spoon)	FisherBrand or equivalent	14-375-10 (Fisher Cat#)
Gloves – Heat Resistant		
Cyanide Manifold	Lachat	10204-00-1-A
Interference filter – 570 nm	Lachat	

- Or equivalent

Table 3: Supplies

Supplies	Manufacturer	Catalog number •
Distillation tubes	Lachat	A17001
Sample Cups 4mL	Westco	927-0018-01
Volumetric Flasks	Fisher	Various
Parafilm	Fisher	
Teflon Chip	Fisher	
• Or equivalent		

10. Reagents and Standards

Table 4: REAGENTS

Reagent	Alias	Concentration	Directions found in:
Potassium Cyanide	Stock Standard	Neat	
Potassium Cyanide	Second Source Stock Standard	Neat	
Potassium Hydroxide		Neat	
Sodium Hydroxide		Neat	
Noah – 0.25 N	Carrier Solution	0.25 N	Table 6
Deionized Water			
Potassium Phosphate, Monobasic, (KH_2PO_4)		Neat	
Sodium dihydrogenphosphate Buffer	Buffer Solution	1 M	Table 6
Chloramine-T hydrate		Neat	
Chloramine-T Reagent	Color Reagent		Table 6
Barbituric Acid		Neat	
Pyridine		Neat	
Hydrochloric Acid		12 M	
Pyridine-Barbituric Acid Reagent	Pyridine-Barbituric Acid Reagent		Table 6
p-dimethylaminobenzalrhodanine	Indicating solution		Table 6
Standard Silver Nitrate Solution		0.0192 N	
Magnesium Chloride Hexahydrate		Neat	
Sulfuric Acid		Concentrated	
Sulfuric Acid/Magnesium Chloride Solution	Releasing Agent	7.11 M H_2SO_4 0.79 M MgCl	Table 6
Nitrate Bismuth		Neat	
Lead Acetate Paper		Neat	
Sulfamic Acid		Neat	
Teflon chips		Neat	

Table 5: STANDARDS

Standard	Concentration	Direction found in:	Alias
Potassium Cyanide	1000 mg/L	Table 7	Stock Standard
Potassium Cyanide Must be different Lot # than Stock	1000 mg/L	Table 7	Second Source Stock Standard
Level 1 Calibration Standard	0.40 mg/L	Table 7	Cal Std. 1
Level 2 Calibration Standard	0.20 mg/L	Table 7	Cal Std. 2
Level 3 Calibration Standard	0.10 mg/L	Table 7	Cal Std. 3
Level 4 Calibration Standard	0.05 mg/L	Table 7	Cal Std. 4
Level 5 Calibration Standard and RLVS Standard	0.02 mg/L	Table 7	Cal Std. 5
Level 6 Calibration Standard	0.00 mg/L	Table 7	Cal Std. 6
Initial Calibration Verification – 1	0.10 mg/L	Table 7	ICV – 1
Initial Calibration Verification – 2	0.05 mg/L	Table 7	ICV – 2
Continuing Calibration Verification	0.20 mg/L	Table 7	CCV
Working Standard (1)	10 mg/L	Table 7	Working Standard (1)
Secondary Working Standard (1)	100 mg/L	Table 7	Secondary Working Standard (1)
Secondary Working Standard (2)	1.2 mg/L	Table 7	Secondary Working Standard (2)
Matrix Spike/Matrix Spike Duplicate – Liquid	0.10 mg/L	Table 7	MS/MSD-Liquid
Matrix Spike/Matrix Spike Duplicate – Solid	0.10 mg/L	Table 7	MS/MSD-Solid
Laboratory Control Spike/Laboratory Control Spike Duplicate - Liquid	0.10 mg/L	Table 7	LCS/LCSD-Liquid
Laboratory Control Spike/Laboratory Control Spike Duplicate - Solid	0.10 mg/L	Table 7	LCS/LCSD-Solid

Table 6: PREPARATION OF REAGENTS

Standard	Alias	Final Concentration	Directions	Final Volume
NaOH – 0.25 N	Carrier Solution	0.25 N	Weigh 10 g of neat NaOH into ~ 500 ml DI water in 1 L plastic container. Dilute to volume. Prepare fresh daily.	1000 ml
Sodium dihydrogenphosphate Buffer	Buffer Solution	1.0 M	Weigh 69 g of neat NaH_2PO_4 into ~ 500 ml DI water in 1 L volumetric flask. Add 2.0 mL of concentrated Probe Rinse Solution. Dilute to volume. Shelf life = 3 months.	500 ml
Chloramine-T Reagent	Color Reagent		Weigh 0.4 g of neat Chloramine-T hydrate into ~ 50 ml DI water in 100 ml volumetric flask. Dilute to volume. **PREPARE DAILY!**	100 ml
Pyridine-Barbituric Acid Reagent	Pyridine-Barbituric Acid Reagent		**MAKE UNDER HOOD!!!!!!!** Weigh 7.5 g barbituric acid into a 500 mL beaker Add 100 ml DI. Add 37.5 ml pyridine, mix to dissolve Add 7.5 ml conc. HCL. Transfer to 500 mL volumetric flask and dilute to volume. Shelf life = 3 months.	500 ml
p-dimethylaminobenzalrhodanine	Indicator Solution		Weigh 20 mg of neat p-dimethylaminobenzalrhodanine into a 100 ml volumetric flask. Dilute into 100 ml acetone. Shelf life = daily/when used.	100 ml
Sulfuric Acid/Magnesium Chloride Solution	Releasing Agent	7.11 M H_2SO_4 0.79 M MgCl	Weigh 110.8 g DI water in beaker. Add 32.2 g Magnesium Chloride Hexahydrate ($MgCl_2.6H_2O$). Slowly add 139 g concentrated H_2SO_4. Transfer to automatic pipette container. Cool. Shelf life = 1 year.	200 g

Table 7: PREPARATION OF STANDARDS

Standard	Alias	Final Concentration	Directions	Final Volume
Potassium Cyanide	Stock Standard.	1000 mg/L	POISON!!!!! Weigh 2.51 g of neat KCN and 2 g of neat KOH into ~900 ml DI H2O in 1 L volumetric flask. Dilute to volume. Shelf life = 3 months. Standardize against AgNO3. If concentration of stock standard is between 995 – 1005 mg/L then use 1ml of stock solution.	1000 ml
Potassium Cyanide	Second Source Stock Standard	1000 mg/L	POISON!!!!! Weigh 2.51 g of neat second source KCN and 2 g of neat KOH into ~900 ml DI H2O in 1 L volumetric flask. Dilute to volume. Shelf life = 3 months. Standardize against AgNO3. If concentration of stock standard is between 995 – 1005 mg/L then use 1ml of stock solution.	1000 ml
Working Standard (1)	Working Standard (1)	100 mg/L	Pipette 10 ml stock standard into 100 ml volumetric flask. Dilute to volume with DI H2O. PREPARE DAILY!	100 ml
Working Standard (2)	Working Standard (2)	1.2 mg/L	Pipette 1.2 ml Working Standard (1) into 100 ml volumetric flask. Dilute to volume with DI H2O. PREPARE DAILY!	100 ml
Secondary Working Standard (1)	Secondary Working Standard (1)	10 mg/L	Pipette 1 mL Potassium Cyanide (second source stock standard) into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. PREPARE DAILY!	100 ml
Calibration standard – All other calibration points are diluted off of this standard by the instrument.	Cal. Std.	0.40 mg/L	Pipette 2.0 mls of Working Standard (2) into 6 ml distillation tube. Dilute to volume with DI H2O. Prepare daily and distill.	6 ml
CRDL Standard	CRDL	0.020 mg/L	Pipette 0.25 mls of calibration standard into 5 ml volumetric flask. Dilute to volume with 0.25N NaoH. Prepare daily.	5 ml
Calibration Blank	Cal. 0	0.000 mg/L	Use 0.25N NaOH only.	100 ml
Continuing Calibration Verification	CCV – 1	0.20 mg/L	Pipette 1.0 ml of Working Standard (2) solution into 6 ml distillation tube. Dilute to volume with DI H2O. Prepare daily and distill.	6 ml
Initial Calibration Verification –1	ICV – A	0.10 mg/L	Pipette 1.0 ml of Secondary Working Standard (2) solution into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare daily.	100 ml
Initial Calibration Verification –2	ICV – B	0.050 mg/L	Pipette 0.50 mls of Secondary Working Standard (1) solution into 100 ml volumetric flask. Dilute to volume with 0.25N NaOH. Prepare daily.	100 ml
Matrix Spike/Matrix Spike Duplicate – Liquid	MS/MSD - Liquid	0.10 mg/L	Pipette 0.50 mls of Working Standard (2) into distillation tubes. Dilute to volume with sample. Prepare daily.	6 ml
Matrix Spike/Matrix Spike Duplicate – Solid	MS/MSD-Solid	0.10 mg/L	Pipette 0.50 mls of Working Standard (2) into sample tubes. Add solid sample. Dilute to volume with DI H2O. Prepare daily.	6 ml
Laboratory Control Spike/Laboratory Control Spike Duplicate – Liquid	LCS/LCSD-Liquid	0.10 mg/L	Pipette 0.50 mls of Working Standard (2) into sample tubes. Dilute to volume with DI H2O. Prepare daily.	6 ml
Laboratory Control Spike/Laboratory Control Spike Duplicate – Solid	LCS/LCSD-Solid	0.10 mg/L	Pipette 0.50 mls of Working Standard (2) into sample tubes. Dilute to volume with DI H2O. Prepare daily.	6 ml

11. Calibration and Standardization

11.1 **Daily Calibration** – The SmartChem must be calibrated each time it is set up and at least every 24 hours that samples are analyzed. Calibration requires analysis of a minimum of 5 standards plus a blank due to the linear regression calibration used. The working range is from the current MDL to the highest standard in the calibration curve.

11.2 **Calibration Verification** – Each calibration must be verified by analyzing an Initial Calibration Verification (ICVA/ICVB) and Initial Calibration Blank (ICB). A Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) must also be analyzed after each set of 10 samples.

11.3 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the Pace Reporting Limit to demonstrate acceptable recovery at the reporting limit.

11.4 **Acceptance Criteria** – The correlation coefficient of the response for elements requiring multiple levels must be ≥ 0.995. The results of the ICV/CCV checks must agree within ± 10%. The results of the ICB/CCBs must be <RL.

11.5 Follow steps in section 12 to run calibration curve.

11.6 The SmartChem can automatically prepare diluted standards and samples, as required, to bring high concentration standards and samples into the working range of the method. See the SmartChem Operation Manual Chapter 3 for instructions on how to configure the SmartChem Parameter Method File.

12. Procedure

NOTE: Document all sample volumes, standards and reagents used in the distillation on the prep log.

12.1 Distillation Procedure - This section of the SOP details the steps in distilling the samples before analysis. Samples must be checked for sulfides before distilling. If sulfides are present and the samples treated with bismuth nitrate, the entire calibration curve must be similarly treated and distilled. Refer to section 6.4 for procedure. In the following procedure, **D** and **M** refer to the marks on the collector tube. **D** means "distillation" end, and **M** means "measuring" end.

12.1.1 Set the controller to 120°C, allow the heater to warm up. The operating temperature of the distillation block needs to be verified annually.

12.1.2 Label tubes with the **M** end up, place as many properly labeled collector tubes as you have samples into the collector tube rack.

12.1.3 For liquid samples, place a drop of sample on lead acetate paper. If the paper darkens this indicates the presence of sulfide. Treat the stabilized sample (pH >12) with bismuth Nitrate. Repeat until a drop on the lead acetate paper does not darken. Filter sample to remove precipitate and use filtrate as sample to be distilled. Place 6 ml of sample into each properly labeled sample tube using a calibrated automatic pipette. Add ~ 0.1 g sulfamic acid.

NOTE: If samples contain and are treated for sulfide, the entire calibration curve must also be similarly treated and distilled along with the samples.

12.1.4 For soil samples, weigh out ~0.20 g of soil or sludge to the nearest 100th on a calibrated balance and dilute to 6 ml with deionized water with a calibrated automatic pipette into each properly labeled sample tube. Add ~ 0.1 g sulfamic acid.

12.1.5 For liquid samples, prepare one method blank by adding 6 mls of DI water to properly labeled sample tube using a calibrated automatic pipette. Add ~ 0.1 g sulfamic acid.

12.1.6 For soil samples, prepare one method blank by weighing out 0.20 g of Teflon chips to the nearest 100th on a calibrated balance and dilute to 6 ml with deionized water with a calibrated automatic pipette into a properly labeled sample tube. Add ~ 0.1 g sulfamic acid.

12.1.7 For liquid samples, prepare one LCS (and one LCSD if needed/requested) by pipetting 6.0 ml of deionized water, ~ 0.1 g sulfamic acid, and 0.50 ml of 1.2 mg/L CN standard into two properly labeled sample tubes using a calibrated automatic pipette. See Table 5.

12.1.8 For soil samples, prepare one LCS (and one LCSD if needed/requested) by weighing out 0.20 g of Teflon chips to the nearest 100th on a calibrated balance. Bring to 6 ml volume with deionized water. Add ~ 0.1 g sulfamic acid and 0.50 ml of 1.2 mg/L CN standard using a calibrated automatic pipette into each properly labeled sample tube. See Table 5.

12.1.9 For liquid samples, select one parent sample and prepare one MS/MSD pair per 10 samples by adding 6.0 ml of sample, ~ 0.1 g sulfamic acid, and 0.50 ml of 1.2 mg/L CN standard into two properly labeled sample tubes using a calibrated automatic pipette. See Table 5.

12.1.10 For soil samples, select one parent sample and prepare one MS/MSD pair by weighing out 0.20 g - 0.50 g of sample to the nearest 100th on a calibrated balance. The sample weight is dependent upon the sample matrix. Bring to 6 ml volume with deionized water. Add ~ 0.1 g sulfamic acid and 0.50 ml of 1.2 mg/L CN standard using a calibrated automatic pipette into each properly labeled sample tube. See Table 5.

12.1.11 Using a calibrated pipette, add 0.75 ml of 7.11 M sulfuric acid / 0.79 M magnesium chloride solution to the sample tube using the supplied automatic pipette.

12.1.12 Immediately push the **D** end of a Cyanide-1 collector tube over the open end of each sample tube to start the seal.

12.1.13 Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing, the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.

12.1.14 The pressing motion should be a smooth constant pressure, which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.

12.1.15 Put on heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.

12.1.16 Set the timer for 30 minutes.

12.1.17 When 30 minutes is up, remove the first tube, use heat-resistant gloves, from the block and immediately pull its sample tube using a downward twisting motion as opposed to a sideways motion. You must remove the sample tube within 4 seconds of removing it for the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution into the 5 gallon waste container.

12.1.18 Invert each collector tube and place it into the collector tube rack, now with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.

12.1.19 Hold collector tube horizontally and rinse its walls with the distillate in order to homogenize it. Roll tube in order to collect all droplets from the walls of the tube. Return the tube to an upright position so the **D** end is up.

12.1.20 With the **D** end up, break the collector tube in half by pulling the **D** end hard toward yourself to break it, the twisting and tearing off the **D** end. Discard the **D** end.

12.1.21 In the **M** end of the tube dilute to the 6.0 ml mark with DI water. This results in the original sample volume, but now in 0.25 M NaOH. Pour in culture tube.

12.1.22 In the event that a distillation needs filtering prior to analysis, the batch QC must also go through the same filtration process. Document in the prep-log.

12.2 Basic System Operation (Analytical) - This portion of the SOP is designed to allow the user to set up and run a method. For a more detailed explanation of the many other options, the user should refer to the SmartChem Software Reference Manual. It is assumed that a method program has already been created for the chemistries you wish to run.

12.3 If not already on, turn on computer, printer, monitor, and the SmartChem instrument. The SmartChem on/off toggle switch is located on the left side of the instrument.

12.4 Log into the PC and Network. Click twice on SmartChem New and enter the username: *SmartChem New WESTCO* and password: *JOE*.

12.5 Build Sequence in LIMSLink of batched samples:

12.5.1 Go into LIMSLink and click on the "*Running Man*".

12.5.2 Select the *Prep* method and click *Start New* followed by *OK*.

12.5.3 In the first two lines enter ICVA and ICVB followed ICB in the third line and CRDL in the fourth.

12.5.4 Click on Get Samples and enter the Batch, the Instrument ID (40WTA9) and click OK.

12.5.5 Repeat the last step for each batch of samples in the sequence.

12.6 SmartChem Sequence Loading and Analysis:

12.6.1 SmartChem Start-up procedure.

12.6.2 Go to Sample Entry

12.6.3 Double click on the desired Method to be run. For cyanide the method is CN-CN 335.4 Cyanide 20 to 400ug/L.

12.6.4 Click on Import from file button

12.6.5 Load Sequence file (create Run Plan) - open the SmartChem folder, scroll all the way to the right, and double click on *SmartChem*.

12.6.6 Save – Click on the red disk to save with the format *CN073112 40WTA9 WETA/EPA335.4 CCR*. 073112 is the month, day, year of the analysis. 40WTA9 is the instrument. WETA is the QUEUE and CCR is an example of analyst initials. Spaces must be used where they are. Click OK.

12.6.7 Save External File by clicking *YES* and Save the run plan as *CN073112*.

12.6.8 Go to System Monitor.

12.6.9 Select and Click on the Run Plan that was created.

12.6.10 Verify that the selected Run Plan is correct.

12.6.11 Load Samples, Standards, Controls, Diluent, and Empty Cups as displayed in the System Monitor.

12.6.12 Check the probe rinse, DI water, and Cleaning Solution bottles.

NOTE: When filling these solutions, pour slowly to minimize foaming. If filling is required while a run is in progress, do not lift siphon tube above the liquid surface. Insert a long stem funnel through the open port in the reservoir cap and fill the container slowly with the respective solution to minimize foaming.

12.6.13 Click Start in System Monitor to display user selectable options before beginning the analysis.

12.6.14 If a Wash Cuvette and/or WBL is required before the start of the analysis, then click on the appropriate button. It is recommended that a Wash Cuvette and WBL be run at the start of each day.

NOTE: It is recommended that a new WBL be run after a Wash Cuvette operation is performed and/or after the removal and/or replacement of the same or a new cuvette into the reaction tray.

12.6.15 Click the Start Button to begin analysis. A box will open. Make sure the Calibration and RBL options are selected for a run that is using a new calibration and click OK.

NOTE: Any daily or periodic maintenance must be recorded in the instrument daily logbook.

12.7 SHUTDOWN:

12.7.1 Click on EXIT icon

12.7.2 Turn off computer, printer and switch on surge suppresser.

13. Quality Control

13.1 Refer to the most current version of SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 **Initial Calibration Verification (ICV)**

13.2.1 The ICVA/ICVB must be analyzed before samples.

13.2.2 The ICVA/ICVB are not distilled.

13.2.3 Concentration must be within ± 10% of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified.

13.2.4 If the ICVA/ICVB is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.2.5 The lot number of the Potassium Cyanide Standard used to make the ICVA/ICVB must be different from that of the calibration curve standards.

13.3 **Continuing Calibration Verification (CCV)**

13.3.1 The CCV is analyzed after every 10 samples.

13.3.2 The CCV is distilled with the batch.

13.3.3 Concentration must be within ± 10% of the true value. When measurements are outside the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified. If the reset CCV fails, recalibrate and reanalyze all samples back to the last acceptable CCV.

13.3.4 If the CCV is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.3.5 The lot number of the Potassium Cyanide Standard used to make the CCV must be different from that of the ICV.

13.4 **Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)**

13.4.1 The ICB must be analyzed after the ICVA/ICVB and before samples. The CCB must be analyzed after the CCV and before samples.

13.4.2 The ICB and CCB are not distilled with the batch.

13.4.3 The absolute value must be < PRL. When measurements are above the PRL, terminate analysis, correct the problem, and the calibration re-verified. If the reset of the I/CCB fails, recalibrate and reanalyze all analytical samples analyzed since the last compliant calibration blank.

13.4.4 If the sample concentration is greater than ten times the concentration in the ICB or CCB, the samples do not need to be reanalyzed.

13.5 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.6 **Method Blank (MB)**

13.6.1 A MB is carried through all prep procedures and analyzed with a frequency of 5% or one per batch of up to 20 environmental samples. The MB must be distilled with batch.

13.6.2 The absolute value must be < PRL.

13.6.2.1 When measurements are above the PRL, terminate analysis, correct the problem, verify the calibration, and reanalyze all analytical samples analyzed since the last compliant calibration blank.

13.6.2.2 If the analyte is detected in the method blank, method blank criteria is evaluated and flagged with an appropriate qualifier to the MDL when MDL reporting is required: qualify with an appropriate qualifier sample results less than 10 times the absolute value detected in the blank. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample-by-sample basis. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

13.7 **Laboratory Control Sample (LCS)**

13.7.1 The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples. A Laboratory Control Spike Duplicate (LCSD) must be analyzed if there is insufficient sample volume to perform a matrix spike/matrix spike duplicate or if the client requests one. The LCS must be distilled with batch.

13.7.2 Concentration must be within $\pm$ 10% of the true value for waters. For soils, the concentration must be within calculated in –house limits or default limits of $\pm$ 20%.

13.7.2.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.2.2 If no errors are found and sufficient sample is available, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still out side the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.7.2.3 If sufficient sample volume is not available, report the sample data with an appropriate (L) qualifier flag on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.7.3 When an LCSD is performed, the precision between the LCS and LCSD must be =/< 20% RPD.

13.7.3.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.3.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.8 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

13.8.1 One pair per up to 10 environmental samples or 10% frequency whichever is more frequent. The MS/MSD must be distilled with batch.

13.8.2 Both QC samples must be calculated for accuracy and precision.

13.8.3 Concentration must be within $\pm$ 10% of the true value for waters. For soils, the concentration must be within calculated in house limits or default limits of $\pm$ 20%.

13.8.3.1 If the four times the concentration of the spike is less than the level of the parent, accuracy need not be calculated.

13.8.3.2 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.3.3 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.8.4 The precision between the MS and MSD must be =/< 20% RPD.

13.8.4.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.4.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.8.5 The parent sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD.

13.8.6 The lot number of the Potassium Cyanide used to make the CCV is different for the MS/MSD.

13.9 The appropriate leach blank must be distilled and analyzed along with the appropriate samples as leached on the same leach date. The absolute value must be < PRL. When measurements are above the PRL, terminate analysis, investigate and correct the problem, verify the calibration, and reanalyze all analytical samples analyzed.

13.10 When preparation of a sample exceeds 14 days past the time of collection, notify the project manager before proceeding. If a sample is run past 14 days after collection, flag the result with an appropriate data qualifier.

13.11 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with an appropriate data qualifier.

13.12 See attachments Table 8 and Table 9 for a summary of QC.

14 Data Analysis and Calculations

14.1 The instrument provides calculated sample results in ug/L. Calculations are only necessary if a dilution was used.

14.1.1 Liquid Calculation

Raw Data Value (ug/L) x Dilution Factor / 1000 = Cyanide (mg/L)

14.1.2 Soil Calculation

$$\frac{\text{Raw Data Value (ug/L) X 0.006 L X Dilution Factor}}{\text{(0.20 \% solids in decimal form x 1000)}} = \text{Cyanide (mg/Kg)}$$

15 Data Assessment and Acceptance Criteria for QC Measures

15.1 See Table 9: Data Assessment

16 Corrective Action for Out-of-Control Data

16.1 See Table 9: Data Assessment

17 Contingencies for Handling Out-of-Control or Unacceptable Data.

17.1 See Table 9: Data Assessment

18 Method Performance

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s (most current revision or replacement). A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

18.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of LOD and LOQ* (most current revision or replacement), for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

18.1.4 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program* (most current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office.

18.1.5 Linear Calibration Range (LCR): The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to ensure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by +/- 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

18.1.5.1 Criteria: All standards must be within +/- 10% of the true value with the exception of the highest standard analyzed over the calibration range that should be outside the 10% rule

18.1.6 Pace Analytical Services, Inc – Green Bay Laboratory will not use any data over the highest calibration standard used. All samples will be diluted and reanalyzed that are over the calibration range.

19 Method Modifications

19.1 Method modifications for EPA Method 9012B and EPA 335.4 are as follows

19.1.1 Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.

19.1.2 All major modification to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.

19.1.3 Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.

19.1.4 EPA Method 9012B is an aqueous method, the laboratory has modified the method to accommodate solid matrices.

19.1.5 EPA Method 9012B and EPA 335.4 are described for macro glassware, the laboratory has chosen to use micro distillation equipment in place of the macro. All sample, reagent and standard volumes have been reduced accordingly

19.2 If a client fails to provide sufficient volume for the method required Matrix Spike/Matrix Spike Duplicate (MS/MSD), the laboratory will analyze a Laboratory Control Spike Duplicate to demonstrate precision. The analytical batch will be qualified with the "M5" data qualifier.

20 Instrument/Equipment Maintenance

20.1 See Section 12.

21 Troubleshooting

21.1 See SmartChem Operating Manual.

22 Safety

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23 Waste Management

23.1 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

24 Pollution Prevention

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

24.3 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25 References

25.1 Pace Quality Assurance manual (most current revision or replacement).

25.2 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" (most current revision or replacement).

25.3 SmartChem 200 Method 280-400E (Rev: April 2007)

25.4 *Micro Dist Method Cyanide-1,* Lachat Instruments, October 12, 2000

25.5 SW846 9012B, Revision 2.0, November 2004.

25.6 EPA 600 R-93-100, Revised August 1993, Method 335.4-1

25.7 Standard Methods for the Examination of Water and Wastewater, 18th Edition, p. 4-18, Methods 4500-CN^- A, B, C, and D (1992)

26 Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

26.1 Table 8: **QC SUMMARY**

26.2 Table 9: **DATA ASSESSMENT/CORRECT ACTION**

26.3 Attachment I: Flowchart

27 Revisions

Revision Number	Reason for Change	Date
S-GB-I-064.Rev.0	First Issue.	19Jan2010
S-GB-I-064-Rev.01	Throughout Document: Updated to new format in SOP: S-GB-Q-017 Preparation of SOPs (most current revision or replacement). Updated SOP References throughout document.	25Sept2012
S-GB-I-064-Rev.02	Throughout Document: Updated to new format in SOP: S-GB-Q-017 *Preparation of SOPs* (most current revision or replacement). Updated SOP References throughout document. Table 7: Updated standard expiration dates. Section 11.1: Updated calibration curve fit to linear regression. Sections 11.2, 13.2 and 13.4: Added language for high/low ICVs (ICVA/ICVB). 12.1.10: Updated Soil weight language. Throughout document: Changed references to sea sand to Teflon chips, updated method references to EPA 9012B.	16Jan2014

Table 8: QC SUMMARY

Analytical Method ⇨ Quality Control Measure ⇩	**EPA 335.4 SW846 9012B** **Frequency**	**Acceptance Criteria**
Method Blank	• One per batch of samples, up to 20 environmental samples, whichever is more frequent. • Must be distilled with batch.	• Project Specific or • Less than the RL (LOWEST STANDARD IN CURVE)
Laboratory Control Spike and Duplicate	• One LCS per batch of samples, up to 20 environmental samples, whichever is more frequent. • An LCSD is required if MS/MSD is not performed or if requested by the client. • Must be distilled with batch.	• Project Specific or • For water 90 –110% with 20% RPD • For solids 80 –120% with 20% RPD
Matrix Spike / Matrix Spike Duplicate	• One pair per batch of samples, up to 10 environmental samples, whichever is more frequent. • Must be distilled with batch.	• Project Specific or • For water 90 –110% with 20% RPD • For solids 80 –120% with 20% RPD
Initial Calibration	• Minimum of 5 standards plus blank. Must be performed every time before samples are analyzed.	• Correlation Coefficient of 0.995
CRDL	• Analyzed after Initial Calibration, but before ICV	• 60-140%
Calibration Verification (ICV/CCV)	• ICV – analyzed after calibration but before samples. **The ICV is not distilled .** • CCV – analyzed after every 10 samples. **The CCV is distilled with batch.**	• Project specific or • Recovery between 90 – 110%
Calibration Blank (ICB/CCB)	• ICB – analyzed after ICV. **The ICB is not distilled with batch.** • CCB – analyzed after every CCV pair. • **The CCB is not distilled with batch.**	• Project specific or • Less than RL (LOWEST STANDARD IN CURVE)
Leach Blank	• The appropriate leach blank must be distilled and analyzed along with the appropriate samples as leached on the same leach date.	• Project specific or • Less than RL (LOWEST STANDARD IN CURVE)

Table 9: DATA ASSESSMENT/CORRECTIVE ACTION

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**If these conditions are not achieved ⇒**
Method Blank and Leach Blanks	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
RLVS standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 times the method blank contamination are reported without qualification.

2. In the absence of project specific or method requirements, in-house generated limits will be used. If the MS or MSD fail because the concentration of the spike is less than 25% of the concentration of the parent, use appropriate flag for the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, use appropriate flag for the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate precision data qualifier. Generate a Non-Conformance Memo.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifier to reflect the deficiencies.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

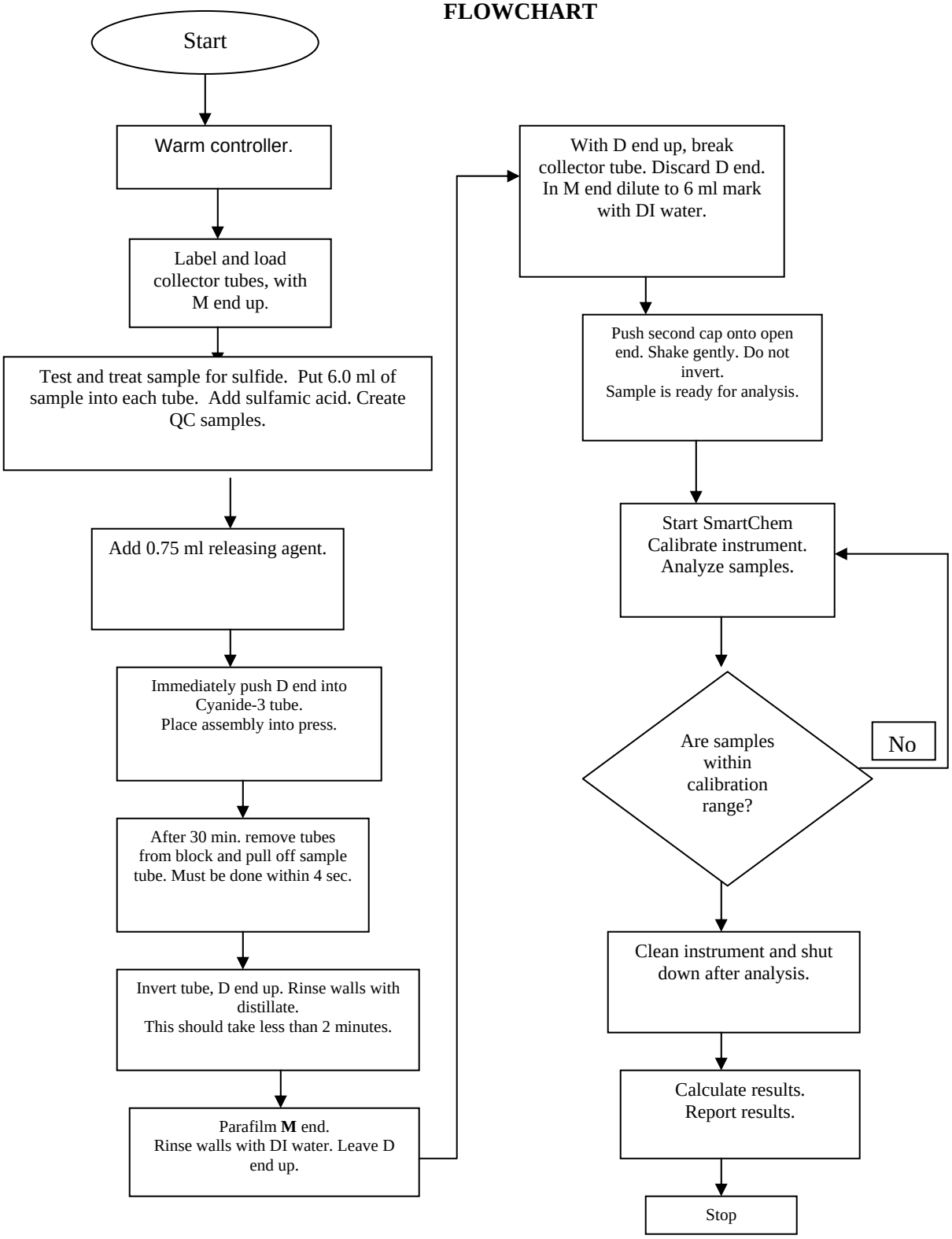
7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB do not have to be reanalyzed.

Table 10: Method Parameters

Type	End Point
Direction	Up
Decimals	2
Model	Linear
Filter 1	570 nm
Sample Blanking	No
Calibration Code	CYN4

Example of Extended Concentration Range 10 to 400 mg/L Without Sample Blanking					
Method Code: WCYN Range: 5-400 ug/L Cn Fluidics - Yes	Volume µL	Delay Time sec.	Read Time sec.	Rinse µL	Code
Sample	150				
Reagent 1 – Sodium Phosphate	63	36	0	0	CNSP
Reagent 2- Chloramine T	15	108	0	0	CNCL
Reagent 3 – Color Reagent	150	0	504	0	CNPY
Maximum Total Reaction Volume	670				
Minimum Sample and Reagent Volume	3				

Attachment I: Flowchart



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STANDARD OPERATING PROCEDURE

Measurement of Volatile Solids and Solids in Waters

Reference Methods: Standard Methods 2540-B, -C, -D, -E, -G, ASTM D2974 and EPA 160.4

SOP NUMBER:	S-GB-I-068-Rev.02
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-068-Rev.01

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/24/13

Kate Grams, Laboratory Quality Manager — Date: 12/17/2013

Chad Rusch, Department Manager — Date: 12/16/2013

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose /Identification of Method

1.1 This Standard Operating Procedure (SOP) describes operations used to measure the parameters in the following table:

Table 1.

Test		Matrix	Method	Reports as
TS	Total Solids	Water	SM 2540B	mg/L
TS	Total Solids	Solid	SM 2540G	%
TSS	Total Suspended Solids	Water	SM 2540D	mg/L
TDS	Total Dissolved Solids	Water	SM 2540C	mg/L
TVS	Total Volatile Solids	Water	EPA 160.4	mg/L
TVS	Total Volatile Solids	Solid	EPA 160.4	%
TVSS	Total Volatile Suspended Solids	Water	EPA 160.4	mg/L
OM	Organic Matter	Solid	ASTM D2974-87(C)	%
FOC	Fractional Organic Carbon	Solid	ASTM D2974-87	%

2. Summary of Method

2.1 Total Solids (TS) – A well-mixed sample is evaporated to dryness and the residue solids are measured gravimetrically.

2.2 Total Suspended Solids (TSS) – A well-mixed sample is filtered. The residue collected by the filter is dried and measured gravimetrically.

2.3 Total Dissolved Solids (TDS) – A well-mixed sample is filtered. The filtrate passing through the filter is evaporated to dryness and the residue solids are measured gravimetrically.

2.4 Total Volatile Solids (TVS, TVSS) – Residue obtained from the determination of TS, or TSS is ignited at 550ºC in a muffle furnace. The loss of weight on ignition is reported as % Volatile Solids for solid samples and mg/L Volatile Solids for liquid samples.

2.5 Fractional Organic Carbon (FOC) - Residue obtained from the determination of TS is ignited at 440ºC in a muffle furnace. The weight loss after ignition is multiplied by a 0.58 factor and reported as % Fractional Organic Carbon.

2.6 Organic Matter (OM) - Residue obtained from the determination of TS is ignited at 440ºC in a muffle furnace. The weight loss after ignition is reported as % Organic Matter.

3. Scope and Application

3.1 This method is applicable to the determination of Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS), and Total Volatile Solids (TVS) in water samples.

3.2 This method is applicable to the determination of Total Solids (TS) and its Volatile (TVS) fractions in soil and semisolid samples. The terms total solids apply to matter left after drying in an oven at 103-105º C and igniting in a muffle furnace at a 550ºC temperature and a defined time interval.

3.3 This method is applicable to the determination of Fractional Organic Carbon (FOC) and Organic Matter (OM) in soil and semisolid samples. The term fractional organic carbon (FOC) applies to the weight loss after ignition, multiplied by a 0.58 factor and reported as % Fractional Organic Carbon.

3.4 This procedure is restricted to use by, or under the supervision of, analysts experienced with the use of laboratory information systems, balances, desiccators, and ovens. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1 This SOP applies to surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, soils, and sludges.

5. Limits of Detection and Quantitation

5.1 The reporting limit (LOQ) for all analytes is listed in Table 1 for the listed methods. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

Table 2.
REPORTING LIMITS

COMPOUND NAME	Aqueous	Solids
	Units mg/L	Units %w/w
	LOQ	LOQ
TS	20	0.1
TSS	10	
TDS	20	
TVS, TVSS	20	
FOC		0.058
OM		0.1

6. Interferences

6.1 Non-representative materials, e.g., leaves and sticks should be removed from the sample prior to measurement unless it is determined that their inclusion is desired.

6.2 Measurements are subject to negative bias for samples containing significant quantities of ammonium carbonate, volatile organics, or other volatile materials that could be lost during drying.

6.3 The residue of samples for TS and TDS that are highly mineralized, especially containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require longer drying, desiccation, and rapid weighing.

6.4 Samples for TS and TDS containing high concentrations of bicarbonate will require careful, and possibly prolonged, drying to ensure that all bicarbonate is converted to carbonate.

6.5 The volumes of aliquots for TS and TDS should be selected to limit the total residue to 200 mg to prevent the residue from crusting over and trapping water during drying.

6.6 Samples for TSS with high TDS, such as saline waters, brines, and some wastes, may be subject to positive bias. Care must be taken to properly rinse the filter to minimize the bias.

7. Sample Collection, Preservation Shipment and Storage

Table 3
SAMPLE COLLECTION, PRESERVATION, HOLD TIME SHIPMENT AND STORAGE

Matrix	Analytical Method	Container(s)	Preservation / Hold Time	Shipment Conditions	Lab Storage Conditions
Aqueous	SM 2540B SM 2540C SM 2540D EPA 160.4	Clean Plastic or glass containers	None / 7 Days	On ice ≤6° Celsius	Walk-in cooler ≤6° C
Soils	SM 2540G ASTM D2974-87	Clean Plastic or glass containers	None / 7 Days	On ice ≤6° Celsius	Walk-in cooler ≤6° C

8. Definitions

8.1 Batch – A grouping of 20, or less, environmental samples of similar matrix, which are prepared and/or analyzed together with the same method.

8.2 Blank Sample – A sample analyzed to determine potential contamination attributed to sample handling and analysis.

8.3 Duplicate Sample – A second aliquot of the same environmental sample analyzed in the same manner as the original sample in order to evaluate precision.

8.4 Environmental Sample – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required.

8.5 Laboratory Control Sample (LCS) – A blank matrix sample spiked with a known concentration of analytes of interest. Laboratory accuracy is evaluated using the LCS.

8.6 Laboratory Information Management System (LIMS) – This is a system for transferring, processing, storing, and reporting analytical results.

8.7 Replicate Samples – Samples collected at the same time, from the same place, for the same analysis, as the original sample in order to determine precision between samples.

8.8 Pace Reporting Limit (PRL) – The lowest level that will be reported by this method. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.

8.9 Solid – matrix such as soils, sediments, sludge, organic liquids, oils or aqueous products and by-products of industrial processes, and aqueous samples with more than 10% settleable solids.

8.10 Total Solids Content (TSC) – Total Solids Content is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature of 103-105ºC.

8.11 Volatile Solids – Residue obtained from the determination of TS is ignited at 550ºC in a muffle furnace. The loss of weight on ignition is reported as % Volatile Solids.

8.12 Refer to Section 10 of the Pace Quality Manual for additional terms used in this SOP.

9. Equipment and Supplies

9.1 Analytical balance – Electronic, capable of weighing to 0.1 mg.

9.2 Vacuum Filtration System – Includes filter holder, membrane filter funnel, vacuum flask and vacuum pump.

9.3 Glass Fiber Filters (for TDS) – GE Water & Process Technologies PreSep Prefiler, Glass, 1.0 Micron, 47mm, or equivalent.

9.4 Glass Fiber Filters (for TSS) – Pre-weighed from Environmental Express, ProWeigh 47mm Glass Fiber Filters, 1.5 µm, with matched aluminum evaporating dishes, or equivalent.

9.5 Graduated Cylinders – 25, 50, 100, 250, 500 and 1000mL.

9.6 Wide bore serological pipettes and pipette helper.

9.7 Drying Ovens – Capable of maintaining 103-105ºC and 178-182ºC.

9.8 Desiccator

9.9 Indicating Desiccant – Drierite Anhydrous Calcium Sulfate, Stock # 2305, or equivalent.

9.10 Laboratory Scoopulas

9.11 Muffle furnace (for volatile solids) – Capable of maintaining temperature of 440ºC and 550ºC.

9.12 Evaporating Beaker (TS/TDS/TVS/OM/FOC) – 50 mL

10. Reagents and Standards

10.1 ERA DataPacK Total Volatile Solids Standard, Cat # 913 for TVS, OM, and FOC analysis, or equivalent.

10.2 ERA QC Plus Solids, Cat #4033 for TS, TSS, and TDS analyses, or equivalent.

10.3 NSI Solutions, Cat # QCI-084 for TS, TSS, and TDS analysis, or equivalent.

10.4 Nanopure Reagent Water – ASTM Type II, or equivalent.

11. Calibration and Standardization

11.1 Balance Calibration – The only required calibration is the analytical balance.

11.2 The balance must be calibrated at least annually by an outside agency and checked **daily** across the range of use, using Class 1 or 2 weights as per the most current version of SOP S-GB-Q-030 *Support Equipment.*

11.3 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

11.3.1 1g for TSS and TVSS with a tolerance of +/-0.0100g

11.3.2 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

12. Procedure

12.1 Create the analytical batch in Horizon.

12.2 Open the Electronic Logbook and create a new worksheet for the batch to be analyzed.

12.3 Pre-Weighed Dried Beaker. Ignite the evaporating beaker at 550ºC for 1 hour in a muffle furnace. Cool in desiccator and store there until ready to use. Record their weight prior to use.

12.4 Check that the oven temperature is within required specifications before placing samples into oven as per the most current version of SOPS-GB-Q-030 *Support Equipment*. Document temperature in the Electronic Logbook.

12.5 **Weighing Dry/Muffle Weights**

12.5.1 Oven temperatures are the same as those described for the test being conducted.

12.5.2 Place the samples back into the oven for 15 minutes and repeat desiccating, cooling, and weighing steps until weight change is $\leq$0.0005g.

12.5.3 If the first and second weights agree within 0.0005 g, or if the weight loss between the first and second weighing is less than 4%, the analysis is complete. If not, place them back into the oven for and additional 15 minutes. Cool in the desiccator, and weigh a third time.

12.5.4 If the third weigh is within 0.0005 g of either the first or second weight or if the weight loss between the second and the third weighing is less than 4%, the analysis is complete.

12.5.5 If the weight loss between the second and third weighing is more than 0.0005g and 4%, heat, cool, and weigh the sample a fourth time and use the fourth weight to calculate the result.

12.6 **Total Solids Determination (TS)**

12.6.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be $\leq$0.0005g before use.

12.6.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water and pour into a tared and dried evaporating beaker. Record the volume for TS-waters and weight for TS-solids analysis in Electronic Logbook.

12.6.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of NSI Solutions (or equivalent) and pour into a tared and dried evaporating beaker. Record the volume for TS-waters and weight for TS-solids analysis in Electronic Logbook.

12.6.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.6.5 Sample

12.5.4.1 For Solid Samples – Weight out 1 to 1.5g of sample in a tared and dried evaporating beaker. Record the weight in Electronic Logbook.

12.5.4.2 For Aqueous Samples - Shake the sample thoroughly and measure an aliquot (between 1mL and 50mL, typically 50mL) into a pre-dried evaporating beaker using a graduated cylinder. Record the volume in Electronic Logbook.

12.6.6 Place the samples to dry in the oven overnight at 103-105°C. (Overnight is a period of time $\geq$ 8 hours.)

12.6.7 After drying overnight, remove from oven and place into the desiccator to cool. After the samples have cooled, obtain the weight of the samples as per Section 13.5, and record in Electronic Logbook.

12.6.8 If Total Volatile Solids (TVS) are also to be performed proceed to Section 12.9. Note: TVS utilizes a different LCS than the TS.

12.7 **Total Suspended Solids Determination (TSS)**

12.7.1 Record Filter IDs and weights in Electronic Logbook for samples and batch QC.

12.7.2 Method blank (MB) – Using a graduated cylinder measure out 1000mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.7.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of Alpha-Trol (or equivalent); record the volume in Electronic Logbook.

12.7.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.7.5 For wastewater, the Pace Analytical Services, Inc. - Green Bay Laboratory uses 500mL in order to meet Wisconsin Reporting Limit requirement of 2.0 mg/L.

12.7.6 For South Carolina work a sample volume is chosen to yield between 2.5 and 200 mg dried residue. If volume filtered fails to meet minimum yield, increase sample volume up to 1 L.

12.7.7 Shake the sample thoroughly and measure the chosen aliquot using a graduated cylinder.

12.7.8 Prepare the filter system with a fresh pre-weighed filter. Handle the filter with forceps only. Wet the filter with a small volume of Nanopure water to seat it. Discard the rinse water.

12.7.9 Filter the sample and wash the filter 3 times with approximately 10mL of Nanopure water. Maintain the filter vacuum for about 1 minute after filtration is complete.

12.7.10 Remove the filter with forceps and place it back into the evaporating dish.

12.7.11 Place the samples (filters and residue) to dry in the oven a minimum of 3 hours at 103-105°C.

12.7.12 After drying, remove from oven and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.7.13 If Total Volatile Suspended Solids (TVSS) are also to be performed proceed to Section 11.10. TVSS utilizes a different LCS than TSS.

12.8 **Total Dissolved Solids (TDS)**

12.8.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.8.2 Method blank (MB) – Using a graduated cylinder measure out 50mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.8.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of APG Solids APG plus Standard (or equivalent), record the volume in Electronic Logbook.

12.8.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.8.5 Shake the sample thoroughly and measure a 50mL aliquot using a graduated cylinder.

12.8.6 Prepare the filter system with a fresh filter. Handle the filter with forceps only. Wet the filter with a small volume of Nanopure water to seat it. Discard the rinse water.

12.8.7 Filter the sample and transfer the filtrate to a pre-weighed dried evaporating beaker.

12.8.1 Place the samples to dry in an oven that has it temperature under a program. The oven starts out at <100 degrees C and increases in temperature according to the program until it reaches 178 - 182 degrees C. It is then held there for a minimum of 1 hour.

12.8.2 After drying overnight, remove from oven and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.9 **Total Volatile Solids (TVS)**

12.9.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.9.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.9.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.9.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.9.5 Sample

12.5.4.3 For Solids Samples – Weight out 1 to 1.5g of sample in a pre-dried, tared evaporating beaker. Record the weight in Electronic Logbook.

12.5.4.4 For Aqueous Samples - Shake the sample thoroughly and measure an aliquot (between 1mL and 50mL, typically 50mL) into a pre-dried evaporating beaker using a graduated cylinder. Record the volume in Electronic Logbook.

12.9.6 Follow TS Section to obtain a dried residue.

12.9.7 Place the evaporating beaker or filter and evaporating dish into the muffle furnace at **550°C** for 1 hour.

12.9.8 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.10 Total Volatile Suspended Solids (TVSS)

12.10.1 Method blank (MB) – Using a graduated cylinder, measure out 1000mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.10.2 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.10.3 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.10.4 Shake the sample thoroughly and measure the chosen aliquot (see sections 11.7.5 and 11.7.6) using a graduated cylinder. Record the volume in Electronic Logbook.

12.10.5 Follow TSS Section to obtain a dried residue.

12.10.6 Place the filter and evaporating dish into the muffle furnace at **550ºC** for 1 hour. Write the filter number on the evaporating dish – paper label will burn off.

12.10.7 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5 in Electronic Logbook.

12.11 Fractional Organic Carbon (FOC) and Organic Matter (OM)

12.11.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be $\leq$0.0005g before use.

12.11.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pour into a tared evaporating beaker, record the weight in Electronic Logbook.

12.11.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker, and record the weight in Electronic Logbook.

12.11.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.11.5 Samples – Into a tared evaporating beaker weigh out about 1-1.5g of sample. Record the weight in Electronic Logbook.

12.11.6 Follow TS Section to obtain a dried residue.

12.11.7 Place the evaporating beakers into the muffle furnace at **440ºC** for 1 hour.

12.11.8 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5 in Electronic Logbook.

12.12 **Entering Results into Horizon**

12.12.1 Post samples and batch QC using Electronic Logbook.

12.12.2 Evaluate batch QC in Horizon.

13. Quality Control

13.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 Method Blank (MB) – A MB is carried through all prep procedures and analyzed with a frequency of 5% or one per batch of up to 20 environmental samples.

13.2.1 TS, TDS, TVS, FOC, and OM all use 50mL of deionized water for the method blank.

13.2.2 TSS and TVSS both use 1000mL of deionized water for the method blank.

13.2.3 For tests that are reported in %, the MB is evaluated in mg/Kg. The mg/kg result must be less than 20 mg/kg. Data can be reported if the sample mg/Kg result is 10x greater than the method blank. The value reported in Epic Pro will be adjusted to 0%.

13.2.4 For tests that are reported in mg/L the absolute value must be < PRL.

13.2.5 When measurements are above the PRL, terminate analysis, correct the problem, verify the calibration, and reanalyze all analytical samples analyzed since the last compliant calibration blank

13.2.6 If the analyte is detected in the method blank between the MDL and the PRL, then samples need to be qualified with a "B" when MDL reporting is required and sample results are greater than the MDL and less than 10 times the absolute value detected in the blank. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample-by-sample basis. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

13.3 Laboratory Control Sample (LCS) – The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples.

13.3.1 ERA DataPacK Total Volatile Solids Standard, Cat. # 913 is used for TVS, FOC, and OM analysis. Equivalent can be used.

13.3.2 APG Solids APG Plus standard, Cat #4033 is used for TDS analysis. Equivalent can be used.

13.3.3 NSI Solutions, Cat # QCI-084 for TS, TSS, and TDS analysis, or equivalent. Equivalent can be used.

13.3.4 A Laboratory Control Spike Duplicate (LCSD) must be analyzed if the client requests one.

13.3.5 The LCS recovered concentration must be within calculated in-house limits or default limits of $\pm$ 20%.

13.3.6 For tests that are reported in %, the LCS is evaluated in mg/L.

13.3.7 When measurements are outside the control limits, check for errors in calculations, standards preparation, and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.3.8 If no errors are found, sufficient sample is available, it is within hold, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still outside the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.3.9 If sufficient sample volume is not available or the samples are now outside of hold, report the sample data with a qualifier flag (L0) on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.3.10 When an LCSD is performed, the precision between the LCS and LCSD must be ≤10% RPD.

13.3.11 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.3.12 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.4 Duplicate – Analyze one sample in duplicate per 10 samples or less. The sample used for the duplicate is selected at random, unless specified by the client. The relative percent different (RPD) between duplicates must be ≤10%.

13.4.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.4.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an "R1" data qualifier

13.5 Constant Weight – Place the samples back into the 103-105ºC oven, and/or 440/550ºC muffle furnace for 15 minutes and repeat cooling, weighing, and desiccating steps until weight change is ≤0.0005g or the weight loss between the two weights is less than 4%.

13.6 Balance – The balance must be verified daily across the range of use, before use, as per the most current version of SOP S-GB-Q-030 *Support Equipment*.

13.6.1 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

13.6.2 1g for TSS and TVSS with a tolerance of +/-0.0100g

13.6.3 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

13.7 Oven – The oven temperature must be within required specifications before placing samples into oven and the temp is recorded in the Electronic Logbook with the correction factor, as per the most current version of SOP S-GB-Q-030 *Support Equipment.*

13.8 Hold – When preparation of a sample exceeds 7 days past the time of collection, notify the project manager before proceeding. If a sample is run past 7 days after collection, flag the result with an "appropriate data qualifier and fill out a LabTrack report.

13.9 See attachments Table A and Table B for a summary of QC

14. Data Analysis and Calculations

14.1 Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS) in mg/L $= \frac{(A - B) \times 1000 \times 1000}{V}$

Where: V = sample volume (mL)

Total Solids	A = final weight (dry weight of residue and dish, g) B = dish weight (g)
Total Suspended Solids	A = final weight (dry weight of residue and filter, g) B = filter weight (g)
Total Dissolved Solids	A = final weight (dry weight of residue and dish, g) B = dish weight (g)

14.2 Volatile Solids (TVS and TVSS mg/L) $= \frac{(A - D) \times 1000 \times 1000}{V}$

14.3 Volatile Solids (TVS %) $= \frac{(A - D) \times 100}{(A - B)}$

14.4 % FOC $= ((1-((D-B) / (A – B))) \times 100) \times (0.58)$

14.5 % OM = ((1-((D-B) / (A-B))) x 100)

Where:

A = dry weight (weight of dried residue and dish, g)
B = dish weight (g)
C = weight of wet sample and dish (g)
D = muffled weight (weight of residue and dish after ignition, g)
V = volume (mL)

14.6 Precision - The relative percent difference (RPD) is calculated as:

$$\textbf{RPD} = \frac{|S - D|}{(S + D)/2} * 100$$

Where: **S** = sample value
D = duplicate value

14.7 Constant Weight is calculated as:

$$\textbf{Constant Weight (\%)} = \frac{|FW - CW|}{(FW + CW)/2} * 100$$

Where: FW = First Weighing, mg

CW = Confirmation Weighing, mg

If FW – CW is 0.0005 g or less, then there is not a need to calculate as a percentage

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 See Section 13 and Table 3: Quality Control, and Table 4: Data Assessment for a listing of Quality Control Criteria.

Table 5.
QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SM 2540G – ASTM D2974-87** **Frequency**	**Acceptance Criteria**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	For solid samples the MB must be < 20 mg/Kg and is reported as 0%. For aqueous samples the MB absolute value must be <RL. Detections > MDL but < RL must be evaluated for data qualification.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. None performed on TVSS.	Recovery must be within +/- 20% of the true value. RPD must be < 10%
Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.	RPD must be < 10%

Table 6.
ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**SM2540G – ASTM 2974-87 If these conditions are not achieved ⇒**
Method Blank	• 1
Duplicate	• 2
Accuracy & Precision Laboratory Control Spikes	• 3

1. In the absence of project specific requirements, sample detects less than 20 times the method blank contamination level are reported with the appropriate data qualifier. Sample detects greater than 20 times the method blank contamination are reported without qualification.
2. In the absence of project specific or method requirements, in-house generated limits will be used. If the DUP fails precision control limits flag the parent with the appropriate data qualifier.
3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate qualifier qualifiers to reflect the deficiencies.

16. Corrective Actions for Out-of-Control Data

16.1 See Section 13 and Table 6.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 If not specifically listed in Section 13 or Table 6, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers.

18. Method Performance

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 Demonstration of Capability (DOC) – Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC). DOC's must be repeated annually. Analyze 4 replicate environmental sample aliquots. Calculate the mean and standard deviations of the results. The relative percent different (RPD) between replicates must be $\leq$ 5% or the study must be repeated.

19. Method Modifications

19.1 Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1 Maintain the analytical balance, ovens, and furnaces according to the most current revision of SOP S-GB-Q-030, *Support Equipment.*

21. Troubleshooting

21.1 Refer to the support equipment operating manual.

22. Safety

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001 *Waste Handling and Management* most current revision or replacement).

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. References

25.1 Pace Analytical Quality Assurance Manual (most current revision or r*eplacement).*

25.2 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" (most current revision or replacement).

25.3 Standard Methods for the Examination of Water and Wastewater, SM Method 2540 B-2011.

25.4 Standard Methods for the Examination of Water and Wastewater, SM 2540 C-2011.

25.5 Standard Methods for the Examination of Water and Wastewater, SM 2540 D-2011

25.6 Standard Methods for the Examination of Water and Wastewater, SM 2540 G-2011.

25.7 ASTM D2974-87, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, May 29, 1987

25.8 VA30007.00.004, Recommendations Regarding the Sampling and Analysis of Fractional Organic Carbon (FOC) in Soils, Technical Decision Compendium, Rule OAC 3745-300-07, March 2000

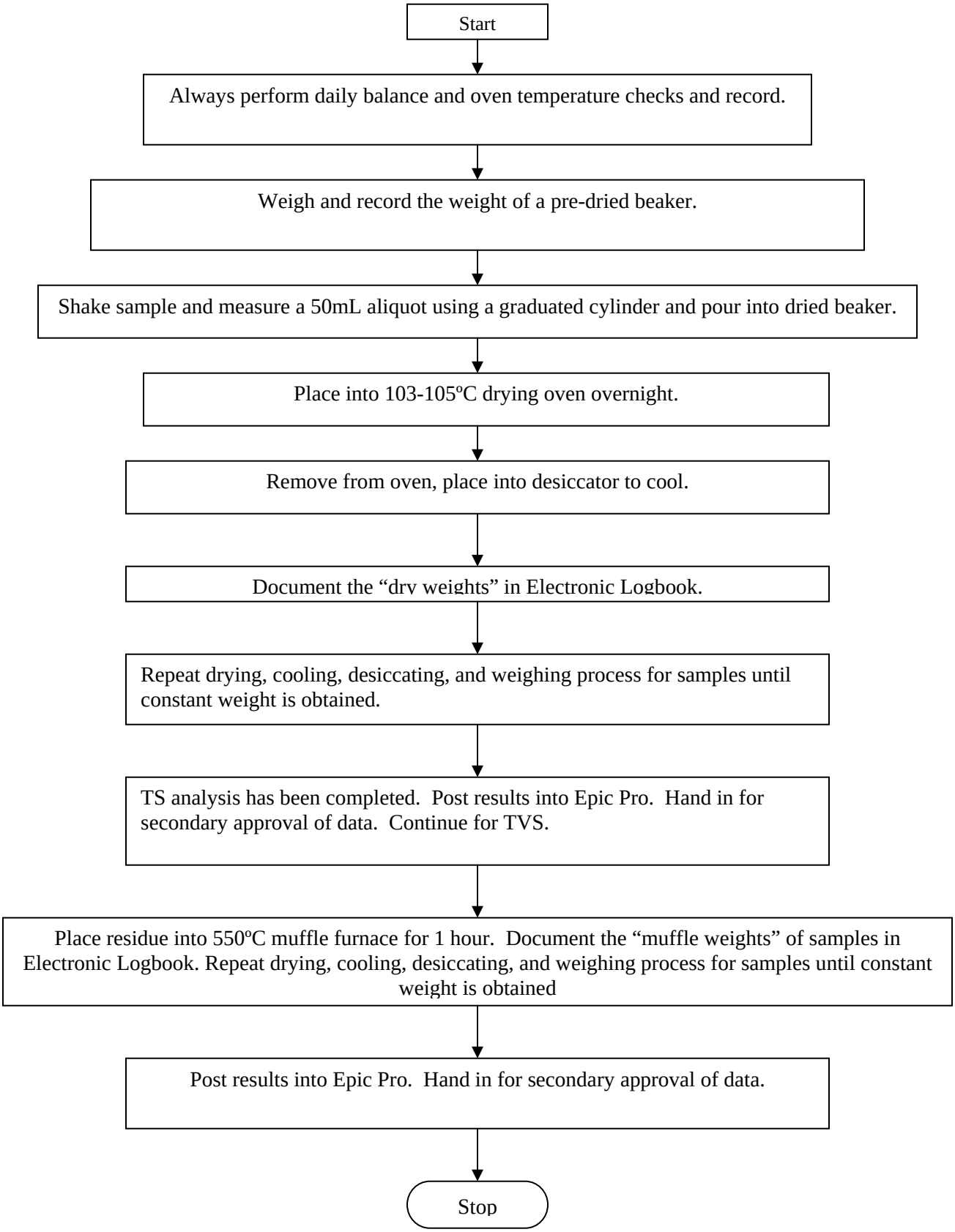
26. Tables, Diagrams, Flowcharts, Appendices, Addenda, etc.

26.1 Flow charts – Attachment I.

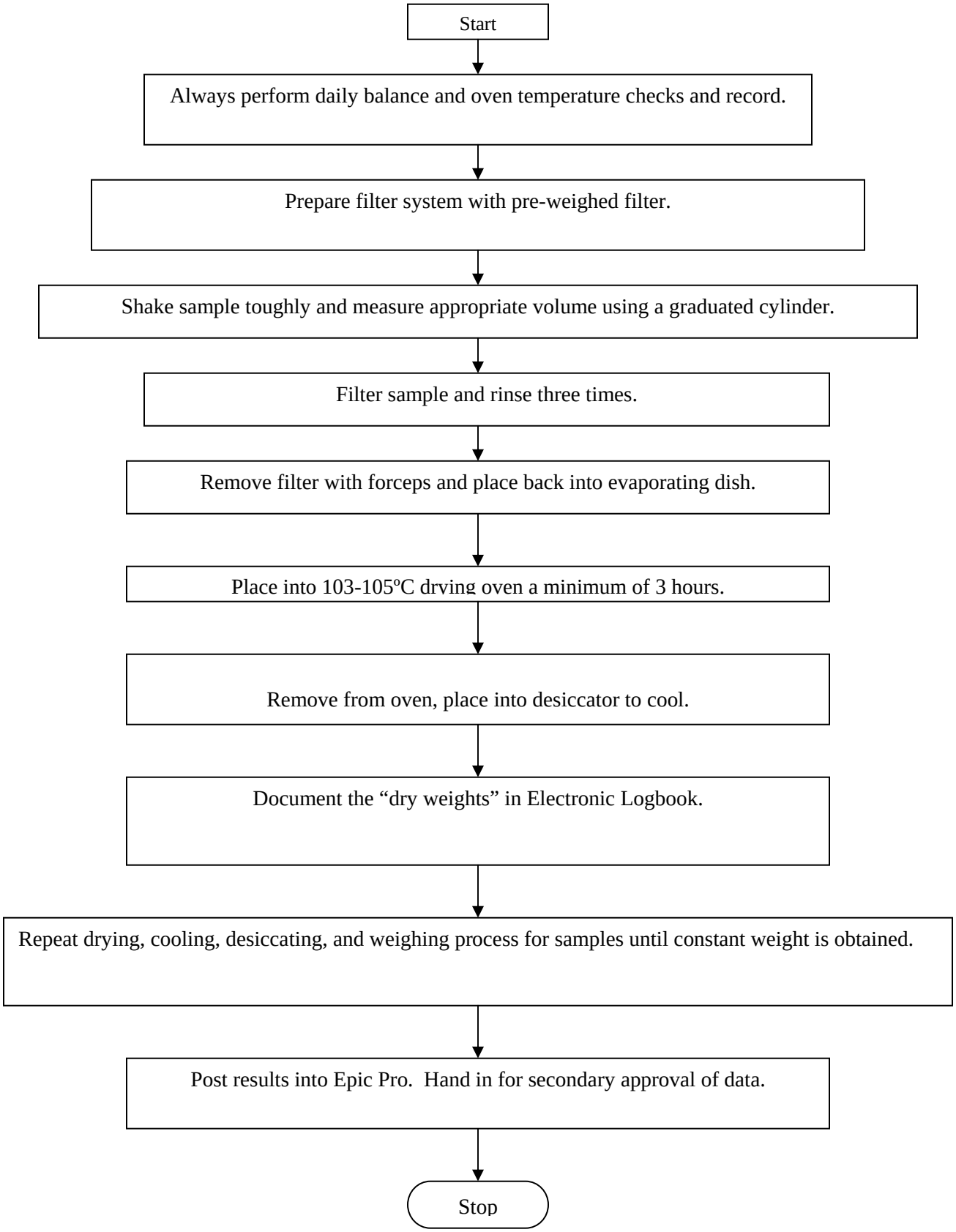
27. Revisions

Document Number	Reason for Change	Date
S-ALL-GB-I-014-Rev.1	Consolidated SOPs ALL-I-014-rev.1, ALL-GB-I-014-rev.1, and GB-I-050-Rev.0. Section 12.6.2 – added South Carolina minimum residue requirements.	24Aug2007
S-ALL-GB-I-014-Rev.2	Corrected FOC and OM calculations and definitions	05Oct2007
S-ALL-GB-I-014-Rev.3	Clarified analysis steps for each test. Changed qualifiers to match Epic Pro.	28Apr2008
S-ALL-GB-I-014-Rev.4	Included notations on how to evaluate MB and LCS for tests reporting in %. Updated Signature Page to Periodic Review.	29May2009
S-GB-I-068-Rev.00	Updated numbering format to match SOP: S-ALL-Q-003, Document Numbering Changed Qualifier from & to L0 and * to R1.	21Sept2010
S-GB-I-068-Rev.01	Updated SOP format to be consistent with SOP: S-GB-Q-017 *Preparation of SOPs* Updated SOP references throughout document. Throughout document: Changed LimsLink to Electronic Prep Log. Sections 2, 3.2, 8.1.2: Removed information relating to Fixed Solids (FS). Sections 2, 3.3, 12.11: Removed information relating to ASH. Section 12.8.1: Changed temperature program to ramp up to 100 deg C. Section 12.10.2: Added LCS requirement to TVSS methods. Tables A and B: Moved and renamed in appropriate sections of SOP.	29Nov2012
S-GB-I-068-Rev.02	Section 25: Updated references to most current revision of the Standard Methods.	16Dec2013

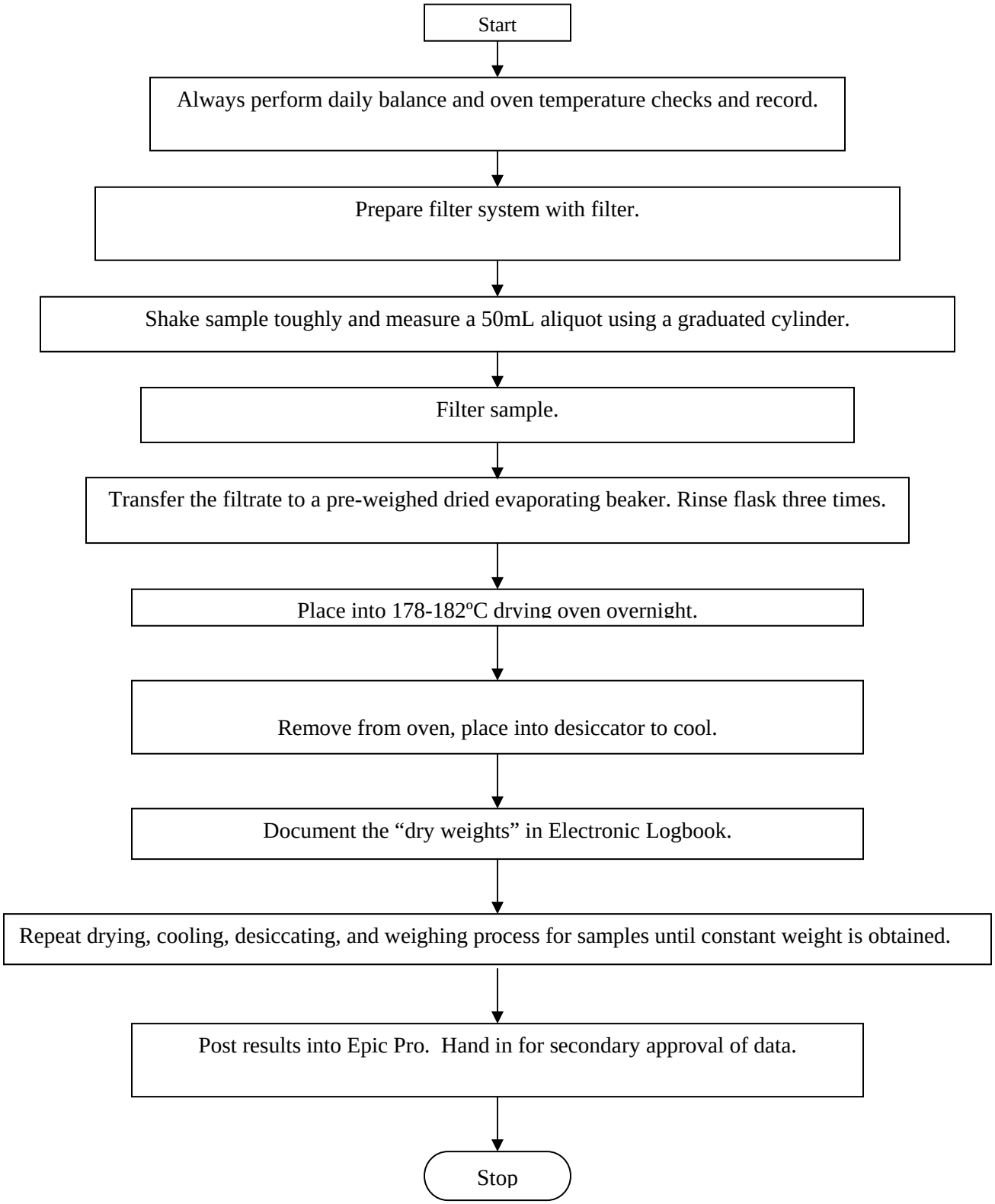
Attachment I – TS and TVS FLOWCHART



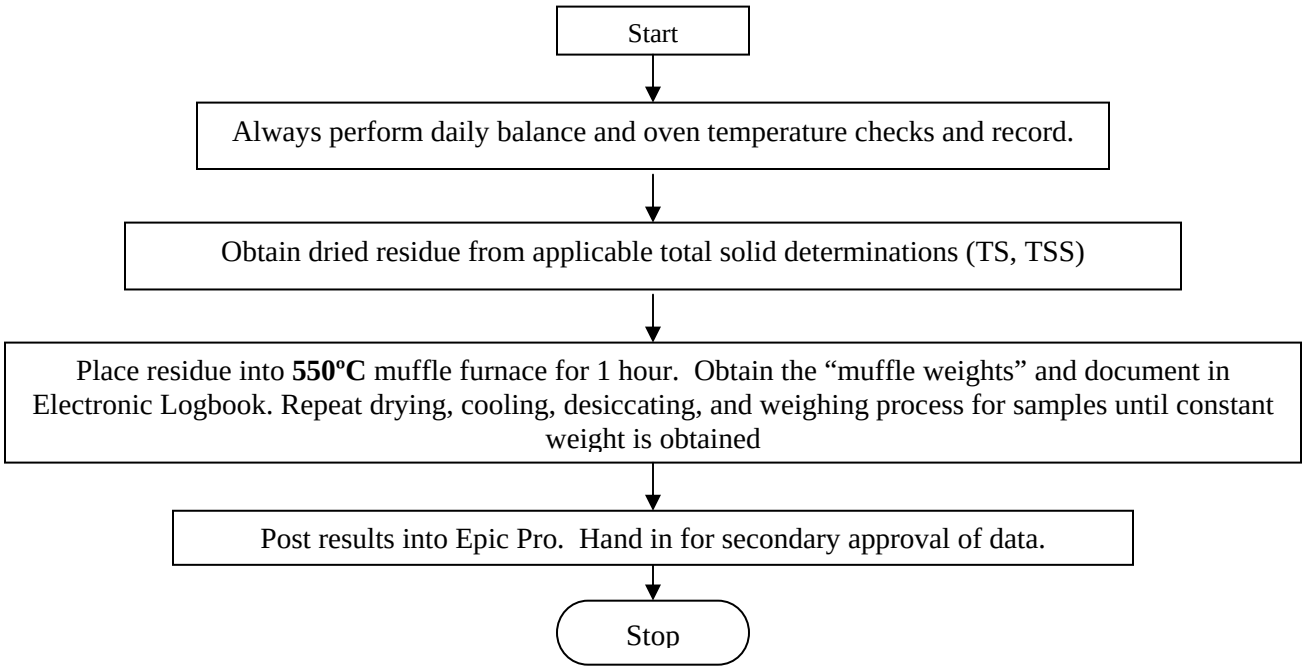
Attachment I – TSS FLOWCHART



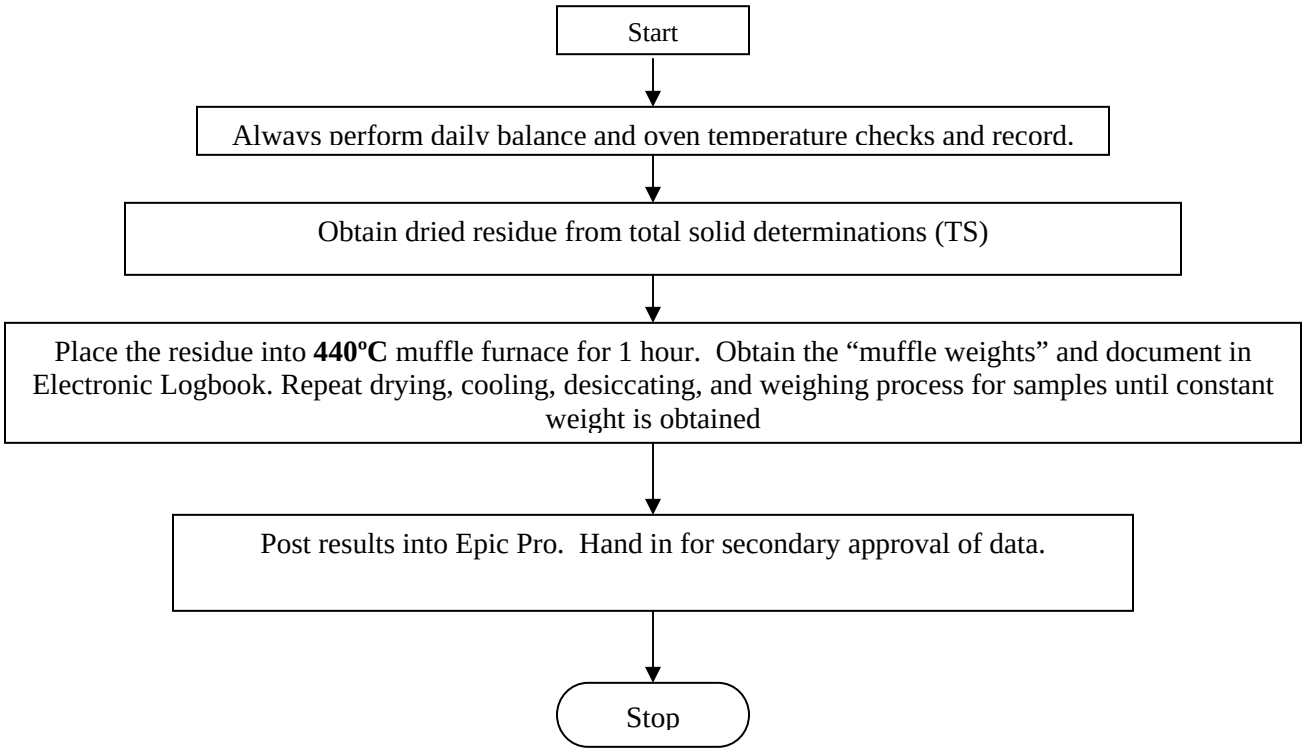
Attachment I – TDS FLOWCHART



Attachment I – TVS and TVSS FLOWCHART



Attachment I – OM and FOC FLOWCHART



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STANDARD OPERATING PROCEDURE

MEASUREMENT OF pH IN WATER, SOIL, AND WASTE

REFERENCE METHODS: SM 4500-H^+ AND SW-846 METHODS 9040B AND 9045C

LOCAL SOP NUMBER:	S-GB-I-071-REV.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S -GB-I-071-REV.00
SOP TEMPLATE NUMBER:	SOT-ALL-I-015-Rev.00

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 09/28/12

Kate Grams, Laboratory Quality Manager — Date: 9/26/12

Chad Rusch, Department Manager — Date: 09/27/2012

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

The purpose of this SOP is to provide a laboratory specific procedure to measure pH in water, soil, and waste samples while meeting the requirements of SM Method 4500-H^+ and SW846 Methods 9040B and 9045C.

2. Summary of Method

2.1. Water samples and wastes containing at least 20% water are measured electrometrically using a combination pH electrode with temperature compensation. The method for analyzing this type of sample is either SM Method 4500-H^+ or SW846 9040B and must be reported referencing one of these methods.

2.2. Soils and solids containing less than 20% water are mixed with reagent water and the pH of the resulting aqueous suspension is measured in the same manner as a water sample. The method for analyzing this type of sample is SW846 9045C and must be reported referencing this method.

3. Scope and Application

3.1. This method is applicable to water samples (including drinking water, surface water, groundwater, saline water, and domestic and industrial wastewaters), soils, sludges, and solid wastes.

3.2. Since pH measurement requires some water content, concentrated acids and bases and concentrated acids and bases mixed with inert substances cannot be measured.

3.3. This SOP was prepared for method-compliant use by all Pace Analytical environmental laboratories.

3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced with electrode measurements. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

4.2. Sodium error at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.

4.3. Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or washing with detergent, then rinsing with distilled water. An additional treatment with hydrochloric acid (1:9) may be necessary to remove any remaining film.

4.4. Sample temperature can cause variations in electrometric measurement of pH in two ways, by causing a change in electrode output and by actually altering the sample pH. Since both variation sources cannot be fully controlled, measurements should be made using a temperature compensating electrode and the analysts should report the sample temperature at the time of analysis along with the pH result. **Coatings of oily material or particulate matter can impair electrode response. The electrode should be thoroughly rinsed and cleaned between samples. Wiping of the electrode using an optical safe wipe may be required to remove oil or clay like soils.**

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves**,** lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

6. Definitions

Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions. In addition to those listed in the QAM, the following are additional terms found in this SOP.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Plastic container (125 mL).	None	If samples cannot be analyzed immediately, they must be stored at 4±2 °C	Samples should be analyzed as soon as possible following collection, preferably at the collection site.(1,2)
Soil	Plastic container (4-oz)	None	If samples cannot be analyzed immediately, they must be stored at 4±2 °C	Samples should be analyzed as soon as possible following collection, preferably at the collection site.(2)

(1) For aqueous samples analyzed and reported according to SM 4500-H^+, PASI observes a 15 minute hold time. Laboratories must qualify results as exceeding the recommended hold time for this ***field parameter*** when analysis occurs beyond this time frame.

(2) For samples analyzed and reported according to either SW-846 9040B or 9045C, PASI observes a 24 hour hold time. Laboratories must qualify results as exceeding the recommend hold time when analysis occurs beyond this time frame.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
pH Meter	Orion	Orion 720A	
Glass Combination Electrode with Automatic Temperature Compensation	Orion	9157BN	Orion Triode pH Electrode
Analytical Balance	Mettler	PJ 480	Capable of weighing to 0.01 g

8.2. General Supplies

Item	Vendor	Model / ID	Catalog #	Description
Disposable Beakers, 50-mL	Fisher		01-29107	
Magnetic Stirrer	Fisher	Corning		
Spin Bars	Fisher			
Wash Bottle	Fisher			
Graduated cylinders	Fisher			50-mL or 100-mL
Laboratory Wipe Tissues	Fisher		06-666A	Kimwipes

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Reagent water	De-ionized water	ASTM Type II water
Electrode Filling Solution	Used for filling electrode	Orion p/n 90004

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, and second source verification.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Primary Calibration Buffer, pH 2.00 ± 0.02	This standard solution is used for daily calibration of the measurement system.	Fisher p/n SB96-1, or equivalent
Primary Calibration Buffer, pH 7.00 ± 0.01	This standard solution is used for daily calibration of the measurement system.	Fisher p/n SB107-4, or equivalent
Primary Calibration Buffer, pH 10.00 ± 0.02	This standard solution is used for daily calibration of the measurement system.	Fisher p/n SB115-4, or equivalent
Primary Calibration Buffer, pH 12.45 ± 0.01	This standard solution is used for daily calibration of the measurement system. Manufactured by Oakton – p/n 00654-12	MG Scientific p/n T-00654-12, or equivalent
Secondary Source Calibration Buffer, pH 4.00 ± 0.01	This standard solution is used to verify calibration before sample analysis.	Fisher p/n SB101-4, or equivalent
Secondary Source Calibration Buffer, pH 7.00 ± 0.01	This standard solution is used to verify calibration before sample analysis. Manufactured by Oakton – p/n 00654-04.	Fisher p/n13300153, or equivalent

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Primary Calibration Buffer, pH 2.00 ± 0.02	Fisher p/n SB96-1, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Primary Calibration Buffer, pH 7.00 ± 0.01	Fisher p/n SB107-4, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Primary Calibration Buffer, pH 10.00 ± 0.02	Fisher p/n SB115-4, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Primary Calibration Buffer, pH 12.45 ± 0.01	MG Scientific p/n T-00654-12, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Secondary Source Calibration Buffer, pH 4.00 ± 0.01	Fisher p/n SB101-4, or equivalent	Manufacturer's recommended expiration date	Store at room temperature
Secondary Source Calibration Buffer, pH 7.00 ± 0.01	Fisher p/n13300153, or equivalent	Manufacturer's recommended expiration date	Store at room temperature

10. Calibration

10.1. Calibration - The measurement system must be calibrated daily with four calibration buffer solutions. These solutions must bracket the range of pH values measured. Additional calibration points are recommended provided the pH meter can accept additional calibration data points.

10.1.1. Press ***power*** to turn the meter on. Press **2nd/channel** until the correct input channel is selected.

10.1.2. Press the **mode** key until the pH mode indicator is displayed.

10.1.3. Press **calibrate.** CALIBRATE is displayed. The time and date of the last calibration is displayed.

10.1.4. After a few seconds ENTER NO. BUFFERS is displayed. Use the numeric keys to enter the number of buffers to be used, then press **yes**. Enter four for the number of buffers to be used.

10.1.5. Place a stir bar in the beaker, put the beaker on the stirrer, and adjust the stirrer to a moderate speed. When the BUFFER 1 prompt appears place electrode(s) in first buffer containing stir bar on.

10.1.6. When the electrode signal has stabilized, the prompt READY CAL AS will appear. Use the numeric keys to enter the value of the buffer. Press **yes** to enter the value.

10.1.7. Rinse the electrode with DI water and gently wipe with a tissue. Repeat steps 5 and 6 for each buffer.

10.1.8. The average electrode slope is displayed at the SLOPE prompt.

10.1.9. The meter automatically proceeds to MEASURE mode.

10.1.10. Record the readings for each buffer used during calibration along with the slope in the pH logbook. (See Attachment I.)

10.2. Initial Calibration Verification – The calibration must be verified with two secondary source calibration buffers prior to conducting measurements on samples. Perform initial calibration verification using the two secondary source calibrations buffers (4.00 and 7.00 pH Units). The control criteria for this verification is listed in Table 12.1.

10.3. Continuing Calibration Verification – The calibration must be verified with a single point verification buffer (pH 7.00) every 10 samples. The control criterion for this verification is listed in Table 12.1.

11. Procedure

11.1. Water Samples

11.1.1. Allow samples to warm to room temperature. Unless a temperature compensated probe is used, samples must be within ±2°C of the standard buffer solutions used for calibration.

11.1.2. Place the sample in a 50-mL disposable beaker, using sufficient volume to cover the sensing elements of the electrode and provide adequate clearance for the magnetic stirring bar. Place a magnetic stirring bar in the beaker and place the beaker on the stirrer. Turn on the stirrer to a moderate speed. The sample should be noticeably mixing but should not develop a vortex.

11.1.3. Rinse the electrode with DI water and gently wipe with a tissue. Place the pH electrode into the sample beaker, taking care to provide adequate clearance for the stirring bar.

11.1.4. Wait for the meter reading to stabilize and note the pH.

11.2. Soil and Solid Samples

11.2.1. Place 20g of sample into a 50-mL disposable beaker. Add 20mLs of DI water and a stirring bar to the beaker and place it on the stirrer. Turn the stirrer to a moderate speed and continuously stir the suspension for 5 minutes.

11.2.2. If the matrix is hydroscopic and has absorbed all the water leaving no remaining free liquid for analysis, repeat the preparation using 20g of sample and 40mLs of DI water.

11.2.3. Turn off the stirrer and allow the sample to sit, permitting the suspended particulates an opportunity to settle to the bottom of the container. Samples may be allowed to sit for up to one hour prior to analysis. Excessive solids in suspension may speed the clogging of the frit between the electrodes.

11.2.4. Place the pH electrode and ATC electrodes into the sample beaker, taking care to immerse the combination electrode just below the suspension. Wait for the meter reading to stabilize and record the pH result and time analyzed. Record the results to 0.1 pH units.

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Secondary Source Calibration Buffers	4.00 and 7.00 pH Units Secondary Calibration Buffer Solutions	Daily after calibration	± 0.1 pH Units	Re-calibrate
CCV Buffer	7.00 pH Units Secondary Calibration Buffer Solution	One after every 10 samples and at the end of the batch of samples.	± 0.1 pH Units	Terminate analysis, correct the problem, re-calibrate the system, and re-analyze the check solutions and all samples that are not bracketed by acceptable QC.
Duplicate Sample	Sample	One per batch of 20 or fewer samples for soil matrices. None required for aqueous samples.	5.0% RPD	Terminate analysis, correct the problem, re-calibrate the system, and re-analyze the check solutions and all samples that are not bracketed by acceptable QC.
Calibration of pH Probe				Calibrate the temperature function of the pH probe on an annual basis as per the current revision of PASI SOP S-GB-Q-030, *Support Equipment.* Record the calibration in the thermometer calibration logbook and label the probe with the date of calibration.

13. Method Performance

13.1. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures* (most current revision or replacement). All results must be in control to qualify the analyst for reporting sample results (within 0.1 pH units of the true value). Results of DOC studies for each analyst shall be retained in the lab quality assurance office. DOCs must be repeated annually. For this test four analysis of the second source pH 7 buffer will serve as the DOCs.

13.2. Method Modifications: If the matrix is hydroscopic and has absorbed all the water leaving no remaining free liquid for analysis, repeat the preparation using 20g of sample and 40mLs of DI water. This is a method modification of 9045C. Method 9045C requires a 1:1 ratio, but since this would not be possible with a sample of this nature the noted procedure will be followed and the sample qualified as such.

14. Pollution Prevention and Waste Management

14.1. Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management* (most current revision or replacement).

14.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15. References

15.1. Method 4500-H^+ B., Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

15.2. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 9040B "pH Electrometric Measurement".

15.3. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 9045C "Soil and Waste pH".

15.4. Pace Analytical Quality Manual; latest revision.

15.5. NELAC Standard; Quality Systems section; latest revision.

16. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

Attachment I - pH Logbook Template.

17. Revisions

Document Number	Reason for Change	Date
SOT-ALL-I-015-rev.00	Restructured document format to new corporate template. Included requirement for duplicate analysis for all aqueous matrices	12Dec2006
S-ALL-GB-I-015-REV.00	Corporate Template updated for use in the Green Bay Laboratory	27Aug2008
S-ALL-GB-I-015_REV.01	Update Signature Page Update Benchsheet	26May2009
S-GB-S-071-Rev.00	Updated SOP numbering following current revision of S-ALL-Q-003 Updated Waste Management and Handling SOP throughout document. Updated pH Logbook to new revision.	27Sep2011
S-GB-I-071-Rev.01	Section 13.2 – Added Method Modification information	26Sept2012

ATTACHMENT I
pH Logbook

Pace Analytical

pH (SW 846 9040B, SW 846 9045C, SM 4500-H+B)

Logbook #

Meter: Orion Model 720A (40WET8)	Meter Serial Number: 008577	
Meter Calibration Date:	Analyst:	
Initial Calibration pH Buffer Solutions		
Value	**pH Readings**	**Reagent Code**
pH 2.00		
pH 7.00		
pH 10.0		
pH 12.45		
Slope:		
Second Source pH Buffer Solutions		
pH 4.00		
pH 7.00		

Sample No.	Date	Time	Matrix	pH.	Adjusted pH	Chlorine	Worksheet #	Analyst

Calibrate pH meter daily with pH buffers 2.0, 7.0, 10.0 & 12.45. Verify the calibration with the second source pH buffers 4.00 and 7.00 (Control Limits +/-0.10 SU). Analyzed second source pH buffer 7.00 after every 10 readings (Control Limits +/- 0.10 SU). Analyze duplicate sample every 20 samples (Control Limits +/- 0.10 SU). For SW846 9040B analyze consecutive measurements of the sample to verify they read within +/- 0.10 SU)

F-GB-I-009-Rev.06 (28Sep2011)

Review/Date_____

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STANDARD OPERATING PROCEDURE

The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy

Reference Methods: EPA SW-846 Methods 7470A and 7471A/B

SOP NUMBER:	S-GB-M-004-REV.04
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-M-004-REV.03

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/19/12

Kate Grams, Laboratory Quality Manager — Date: 12/7/2012

Chad Rusch, Department Manager — Date: 12/07/12

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE

1.1 The purpose of this Standard Operating Procedure (SOP) is to describe the determination of mercury in water, soil/solid samples, and biological tissue.

2. SUMMARY OF METHODS

2.1 Cold vapor atomic absorption utilizes the volatile property of elemental mercury at the 253.7 nm wavelength. To release mercury from organic complexes, the sample is digested with oxidizing reagents and acids in a hot block digester. After digestion, the oxidizing reagents are neutralized. Stannous chloride is added to reduce ionic mercury to the ground state. The Flow Injection Analysis System sweeps the volatile elemental mercury out of the sample and into the cell of an atomic absorption spectrophotometer. The absorbance signal is proportional to the amount of mercury in the sample.

3. SCOPE AND APPLICATION

3.1 This SOP describes the measurement of mercury from the MDL and Reporting Limit to the highest point of the calibration curve.

3.2 This procedure is restricted to use by, or under the supervision of, analysts experienced in the digestion of samples for metals analysis and analysis of digestates by atomic absorption spectrometry. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. APPLICABLE MATRICES

4.1 This SOP is applicable to water samples (including drinking water, surface water, and domestic and industrial wastewaters), solid samples (including soils, sediments, sludges, and, solid wastes), wipes, TCLP leachates, and biological tissue samples.

5. LIMITS OF DETECTION AND QUANTITATION

5.1 Current MDL and RL can be found in the Laboratory Information Management System (EpicPro).

6. INTERFERENCES

6.1 Biota samples can contain diverse matrix types, each of which may present analytical challenges. Spiked samples and Laboratory Control Samples are important for determining digestion efficiency.

6.2 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide, as NaS, do not interfere with recovery of added inorganic Mercury from D.I. water.

6.3 Copper has also been reported to interfere; however, copper concentrations as high as 10mg/L had no effect on recovery of Mercury from spiked samples.

6.4 Samples high in chlorides require up to 7.5 mL of additional potassium permanganate. During the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the Mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent. Samples may be diluted to decrease the chlorides. Alternatively, the sample may be allowed to stand for at least an hour under a hood (without active purging) to remove the chlorine.

6.5 Certain volatile organic materials that absorb at this wavelength may also interfere. A preliminary run without reagents would determine if this type of interference is present.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 BIOTA SAMPLES

7.1.1 Biota samples can be collected in clean plastic or glass containers or plastic baggies.

7.1.2 Biota samples are kept frozen until time of preparation to preserve integrity. Hold time is 28 days from removal from freezer.

7.2 SOIL SAMPLES

7.2.1 Soil and waste samples should be collected in clean wide-mouth glass containers to facilitate obtaining representative aliquots for measurement.

7.2.2 Samples should be stored at 4±2°C and analyzed as soon as possible to minimize microbiological decomposition of organic solids. Samples must be analyzed within 28 days of collection.

7.3 WIPE SAMPLES

7.3.1 Wipe should be collected in clean wide-mouth glass containers.

7.3.2 Samples should be stored at 4±2°C and analyzed as soon as possible to minimize microbiological decomposition of organic solids. Samples must be analyzed within 28 days of collection.

7.4 WATER SAMPLES

7.4.1 Water samples may be collected in plastic or glass containers and must be preserved with HNO_3 to pH <2 and stored at room temp, acid not to exceed 5% of the container capacity.

7.5 SAMPLES MUST BE ANALYZED WITHIN 28 DAYS OF COLLECTION

NOTE: Aqueous samples that react violently to the addition of acid may be collected without chemical preservation with proper variances approved by the regulatory authority. The responsibility of requesting this variance lies with the sample collector.

NOTE: Samples may be preserved in the lab. The samples may not be processed until 24 hours after preservation with a pH test of <2.

8. DEFINITIONS

8.1 Definitions of terms found in this SOP can also be found in the Pace Quality Manual. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP or the Pace Analytical Services' Quality Manual Glossary, Section 10.0.

8.2 Biota Control Blank (Matrix Blank) - A sample of a matrix that is used for the control spike. The biota control blank will either be catfish, tilapia, chicken, or other tissue whichever is available at the time of analysis. The biota control blank should be "farm-raised" to minimize background mercury levels. The concentration in the biota control blank will be subtracted from the concentration of the laboratory control spike. This is done because the biota control blank is known to have some contamination. This SOP will reference biota control blank for ease. Note: For plant material analysis, alfalfa can be used as the biota control blank matrix.

8.3 Reagent Grade – Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents, which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

8.4 Standardized Reference Material (SRM) – A certified reference material produce by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. A SRM is analyzed with each analytical batch of biota samples.

8.5 Pace Reporting Limit (PRL) – The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e. statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.

9. EQUIPMENT AND SUPPLIES

9.1 Heating Block – Operated at 95 $\pm$ 5°C. Heating block should be Environmental Express "Hot Block", or equivalent.

9.2 Digestion Vials – Pre-cleaned 68- mL screw cap vials, Environmental Express (SC475), or equivalent.

9.3 Analytical Balance – Capable of weighing to 0.01g.

9.4 Volumetric Flasks – Assorted, Class A

9.5 Graduated Cylinder – 50-mL

9.6 Mechanical Pipettes – Assorted adjustable air-displacement pipettes with disposable tips (Eppendorf or equivalent).

9.7 pH Strips

9.8 CVAA-FIMS with auto-sampler

9.9 Inert Sparging and Carrier Gas – Argon

9.10 Auto-sampler vials

9.11 Auto-sampler Pump Tubing

9.12 Specimen Cups

9.13 DOLT-4

9.14 Analytical Balance – Capable of weighing to 0.0001g.

10. REAGENTS AND STANDARDS

10.1 Trace Metals Water

10.2 Nitric Acid (HNO_3) – Trace metals grade

10.3 Hydrochloric Acid (HCl) – Trace metals grade

10.4 Sulfuric Acid (H_2SO_4) – Trace metals grade

10.5 Sodium Chloride (NaCl)

10.6 Potassium Permanganate ($KMnO_4$) solution (5%) – Dissolve 50.0 g of $KMnO_4$ into ~800 mL of trace metals water. Dilute to 1000 mL. Prepare fresh each month.

10.7 Potassium Persulfate ($K_2S_2O_8$) solution (5%) – Dissolve 50.0 g of $K_2S_2O_8$ into ~800 mL of trace metals water. Dilute to 1000 mL. Warm to dissolve. Prepare fresh each month.

10.8 Hydroxylamine Hydrochloride ($NH_2OH \bullet HCl$) Solution (12%) – Dissolve 120 g NaCl and 120g $NH_2OH \bullet HCL$ in ~800 mL of trace metals water. Dilute to 1000 mL. Prepare fresh each month.

10.9 Stannous Chloride Solution ($SnCl_2$) - Dissolve 11.0 g of SnCl in ~ 800 mL of trace metals water. Add 30 mL of HCL. Dilute to 1000 mL. Prepare fresh every other day.

10.10 Analytical Standards – Standard solutions are required calibration, calibration checks, and sample spiking solutions. The following describes the contents of each type of solution. The commercial sources for stock solutions, recipes for preparing dilutions and working standards, and concentrations in standard solutions are presented in Table A, Table B, and Table C.

Table A
Stock Standards

Standard	Concentration	Vendor	Catalog #
Hg Calibration Stock	1000µg/mL	High-Purity Standards	CGHG1-1
ICV Calibration Stock	1000µg/mL	Inorganic Ventures, Inc.	100033-1

Standards are used until they expire based on the manufacturer's expiration date.

Table B
Intermediate and Calibration Standards
Waters

Standard	Stock Standard	Conc.	Amount Used	Amount of conc. HNO_3 used	Final Volume (with trace metals water)	Final Conc.
Hg ICV Stock	-	1000µg/mL	-	-	-	1000 µg/mL
Hg Calibration Intermediate	Hg Calibration Stock	1000µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg ICV Intermediate	Hg ICV Stock	1000 µg/ml	20 µL	6.0 mL	200 mL	100 ppb
Hg Cal. 1	-	-	-	-	20 mL	0.0 ppb
Hg Cal. 2 and PRLs	Hg Cal. Intermediate	100 ppb	40 µL	-	20 mL	0.2 ppb
Hg Cal. 3	Hg Cal. Intermediate	100 ppb	200 µL	-	20 mL	1.0 ppb
Hg Cal. 4	Hg Cal. Intermediate	100 ppb	500 µL	-	20 mL	2.5 ppb
Hg Cal. 5	Hg Cal. Intermediate	100 ppb	1000 µL	-	20 mL	5.0 ppb
Hg Cal. 6	Hg Cal. Intermediate	100 ppb	2000 µL	-	20 mL	10.0 ppb
Hg ICV	Hg ICV Intermediate	100 ppb	800 µL	-	20 mL	4.0 ppb
Hg CCV	Hg Cal. Intermediate	100 ppb	1000 µL	-	20 mL	5.0 ppb
Hg ICB/CCB	-	-	-	-	20 mL	0.0 ppb

All intermediate dilution solutions must be prepared as needed, or at least every 7 days. Working solutions must be prepared fresh daily. Solutions may be stored at room temperature.

Table C
Intermediate and Calibration Standards
Soils/Biota

Standard	Stock Standard	Conc.	Amount Used	Amount of conc. HNO_3 used	Final Volume (with trace metals water)	Final Conc.
Hg ICV Stock	-	1000µg/mL	-	-	-	1000 µg/mL
Hg Calibration Intermediate	Hg Calibration Stock	1000µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg ICV Intermediate	Hg ICV Stock	1000 µg/ml	20 µL	6.0 mL	200 mL	100 ppb
Hg Cal. 1	-	-	-	-	30 mL	0.0 ppb
Hg Cal. 2 and PRLS	Hg Cal. Intermediate	100 ppb	60 µL	-	30 mL	0.2 ppb
Hg Cal. 3	Hg Cal. Intermediate	100 ppb	300 µL	-	30 mL	1.0 ppb
Hg Cal. 4	Hg Cal. Intermediate	100 ppb	750 µL	-	30 mL	2.5 ppb
Hg Cal. 5	Hg Cal. Intermediate	100 ppb	1500 µL	-	30 mL	5.0 ppb
Hg Cal. 6	Hg Cal. Intermediate	100 ppb	3000 µL	-	30 mL	10.0 ppb
Hg ICV	Hg ICV Intermediate	100 ppb	1200 µL	-	30 mL	4.0 ppb
Hg CCV	Hg Cal. Intermediate	100 ppb	1500 µL	-	30 mL	5.0 ppb
Hg ICB/CCB	-	-	-	-	30 mL	0.0 ppb

All intermediate dilution solutions must be prepared as needed, or at least every 7 days. Working solutions must be prepared fresh daily. Solutions may be stored at room temperature.

11. CALIBRATION AND STANDARDIZATION

11.1 A digested calibration curve is made up every time there are samples prepared. The curve is associated to the sample batch it is prepped with. Calibration requires analysis of a calibration blank and at least five levels of calibration solutions. The lowest calibration concentration is the Pace Reporting Limit.

11.2 The resultant correlation coefficient must be greater than 0.995. If this does not occur, then the instrument must be recalibrated and or the calibration curve and the associated samples must be re-prepped.

11.3 The calibration curve must pass an Initial Calibration Verification (ICV) that is analyzed after the calibration standards and before any samples. The ICV concentration is near the midpoint of the calibration curve and is made from a source other than the one used to make the calibration standards. If the ICV is outside the control limits, reanalyze the ICV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. All samples must be reanalyzed.

11.4 Pace Reporting Limit Standard (PRL Std) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

11.5 Every ten samples or less must be bracketed by Continuing Calibration Verifications (CCV). The CCV concentration is near the midpoint of the calibration curve. The CCV limits are +/- 10% of their expected values. If a CCV is outside the control limits, reanalyze the CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed.

11.6 An acceptable Initial Calibration Blank (ICB), must be analyzed after the ICV.

11.7 Continuing Calibration Blanks (CCB) are analyzed after the CCVs.

11.8 The control limit for the ICB and CCB is less than the Pace Reporting Limit. If an ICB/CCB is outside the control limits, reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed unless the sample concentration is $\geq$10X the blank detection or the sample is an ND for blank failures that have a + bias.

12. PROCEDURE

12.1 Sample Preparation

12.1.1 Water samples

12.1.1.1 Verify preservation in the samples by checking their pH with a test strip (done in Sample Receiving).

12.1.1.2 Prior to analysis, the samples and the calibration curve must be digested.

12.1.1.3 Turn on the heating block and set the temperature to maintain a sample temperature of 95$\pm$5°C. Before placing samples in the block to digest, verify that the sample well temperature is correct and

adjust as required. Record the temperature in the Electronic Preplog. Verify that the hood is functioning.

12.1.1.4 Add 20 mL of trace metals water and the appropriate amounts (as shown in Table 2) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.

12.1.1.5 Add 20 mL of trace metals water to a digestion vial for the Method Blank.

12.1.1.6 Add 20 mL of trace metals water and 1.0 mL of Hg ICV Intermediate to two digestion vials for the Laboratory Control Spike and Laboratory Control Spike Duplicate. Only prep LCSD if requested by client or there is not enough sample to run an MS/MSD.

12.1.1.7 Add 20 mL of each well-mixed sample to digestion vials.

12.1.1.8 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random or assigned by the client. An additional 20 mL of this sample along with 1.0 mL of Hg ICV Intermediate is added to two digestion vials.

12.1.1.9 To each digestion vial add 0.5 mL of concentrated nitric acid. Swirl the digestion vials gently.

12.1.1.10 To each digestion vial add 1.0 mL of concentrated sulfuric acid. Swirl the digestion vials gently.

12.1.1.11 Add 3 mL of 5% $KMnO_4$, , swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, an additional 3 mL of $KMnO_4$ solution is added to all samples, QC, and standards. **Note: If the sample still does not maintain color, discard applicable samples and dilute the samples prior to digestion.**

12.1.1.12 Add 1.6 mL of 5% Persulfate solution. Place lid or cap on tube so as to allow pressure to vent but minimizing evaporation. Swirl the digestion vials gently.

12.1.1.13 Digest samples for 2 hours at 95±5°C.

12.1.1.14 Remove samples and let cool to room temperature.

12.1.1.15 Add 1 mL of 12% hydroxylamine hydrochloride solution. Swirl the digestion vials gently.

12.1.1.16 Samples are now ready for analysis.

12.1.2 Soil/Solid/Biota/Wipe Samples

12.1.2.1 Prior to analysis the samples and calibration curve must be digested.

12.1.2.2 Turn on the heating block and set the temperature to maintain a sample temperature of 95±5°C. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the temperature in the Electronic Preplog. Verify that the hood is functioning.

12.1.2.3 Add 30 mL of 3% HNO_3 and the appropriate amounts (as shown in Table 2) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.

12.1.2.4 Prepare one Method Blank by adding glass beads to it. For biota analysis, leave the Method Blank empty. A Biota Control Blank is also prepared by weighing 0.6 g of homogenized biota control blank tissue into a digestion vial.

12.1.2.5 Prepare the Laboratory Control Spike (LCS) for soils, wipes, and wastes by adding glass beads to it. Then, add 1.5 mL of Hg ICV Intermediate to the digestion vial. Prepare the LCS for biota samples by weighing 0.6 g of homogenized biota control blank into the digestion vial. Add 1.5 mL of Hg ICV Intermediate the vial. Only prep a Laboratory Control Spike Duplicate (LCSD) if requested by client or there is not enough sample to run an MS/MSD. Prepare the LCSD in the same fashion as the LCS.

12.1.2.6 For biota samples weigh approximately 0.05 g of Standard Reference Material on an analytical balance (must read to at least 4 places past the decimal) into a labeled digestion vial.

12.1.2.7 Weigh 0.6 g of homogenized sample into a labeled digestion vial. Record the weight to the nearest 0.01 g in the digestion logbook. For samples with high liquid content, a larger sample size may be used, as long as the digestion is complete. For wipes, put the WHOLE wipe in the digestion vial.

12.1.2.8 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random. Weigh 2 additional 0.6 g aliquots into digestion vials. Add 1.5 mL of Hg ICV Intermediate to each vial.

12.1.2.9 Add 30 mL of 3% HNO_3 solution to all QC and samples. Swirl digestion vials to gently mix.

12.1.2.10 To each digestion vial add 0.5 mL of concentrated nitric acid. Swirl digestion vials to gently mix.

12.1.2.11 To each digestion vial add 2.0 mL of concentrated hydrochloric acid. Swirl digestion vials to gently mix.

12.1.2.12 Add 3 mL of 5% $KMnO_4$, place lid or cap on tube so as to allow pressure to vent but minimizing evaporation, swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, an additional 3 mL of $KMnO_4$ solution is added to all samples, QC, and standards. **Note: If the sample still**

does not maintain color, discard set and dilute the sample prior to digestion. For Biota samples, add 6ml of KMnO4 to start with.

12.1.2.13 Digest samples for 30 minutes at 95±5°C.

12.1.2.14 Remove samples and let cool to room temperature.

12.1.2.15 Add 3 mL of 12% hydroxylamine hydrochloride solution. Swirl digestion vials to gently mix.

12.1.2.16 Samples are now ready for analysis.

12.2 Basic System Operation – Analytical

12.2.1 This portion of the SOP is designed to allow the user to set up and run a method, print a sample report, then shut down using the more basic software functions. For a more detailed explanation of the many other options, the user should refer to the Reference Manual.

12.2.1.1 Verify the carrier argon gas supply is on.

12.2.1.2 Switch on the FIMS 100.

12.2.1.3 Verify the computer is on. If not switch on the computer and enter network password and click on Start.

12.2.1.4 Go to programs, FIMS, and click on the AA WinLab Analyst and choose a custom workspace.

12.2.1.5 Click on GBWRK.FMS and then OK.

12.2.1.6 Go to “Sample Information Editor”, File, Open Sample info File.

12.2.1.7 Choose the most recent applicable file and click OK.

12.2.1.8 Make changes to the calibration curve IDs in locations 1-9 and enter sample information starting with Autosampler Location #10. Enter the LIMS ID numbers under sample ID columns.

12.2.1.9 Go to File, Save As and change the Sample Info File identification to match the data file name and click OK.

12.2.1.10 In the menu click on Print and sample information to get a copy of the sample information you just entered to check for errors.

12.2.1.11 Close the window by clicking on the X in the upper right-hand corner.

NOTE: Only sample information should be entered in this file. Information on blanks, standards and QC samples is entered in the QC page of Method Editor.

12.2.1.12 Under the QC page you set up the QC sample ID, i.e. ICVs and CCVs and the autosampler location. Set the QC concentration and the control limits.

12.2.1.13 On the Schedule Timing page set up when the QC will be analyzed, i.e. after calibration, after 10 samples, and at the end. IF re-calibration occurs the QC must be analyzed at the same frequency.

12.2.1.14 Under the Actions Calib. And Periodic page, specify what to do if calibration QC fails, for example recalibrate, recalibrate and rerun, or stop.

12.2.1.15 On the Actions: End and Retry page, specify what QC to run at the end of a run and corrective action if QC fails.

12.2.1.16 Under the Options page, set how you want data printed (usually default). It is also possible to specify saving the peaks and add comments about the run.

12.2.1.17 In the Automated Analysis Window there are 2 pages: Set UP, and Analyze.

12.2.1.18 Under "Use Autosampler Locations listed Below", type in the numbers or range of numbers to be analyzed.

12.2.1.19 Select the name of the Results Data Set to save the results. Naming example: 0412101, denotes the, month, day and year. A number is used for describing each run within a day's output.

12.2.1.20 The Save Data box and Print Log box should already be selected.

12.2.1.21 Off After Analysis. Pumps should be checked.

12.2.2 Starting the Analysis

12.2.2.1 Prepare the required reagents: Carrier solution and reducing agent.

12.2.2.2 Clamp down the tubes by swinging the pressure levers until they lock. Place inlets of each solution in proper container: Yellow/blue in the carrier and red/red in the reductant. Click on the FIAS icon to turn on the pump to draw solutions through the tubing. Check on the flow.

12.2.2.3 Click on the Analyze All button. The system will run the blank and standards specified in the Element Parameter file, then the entire sample specified in the Sample Info. File. Check the calibration curve by clicking on Calibration icon on the tool bar.

12.2.2.4 To run the blank and standards only: In order to check the calibration curve before running any samples:

12.2.2.5 Click on Calibration, the system will run the blank and standards specified in the Element File and complete the calibration curve. To check the calibration curve, click on the Calibration icon on the toolbar.

12.2.2.6 To continue the run, click on the Auto control window then click on Analyze sample. The system will run the samples specified in the Sample Information file.

12.2.2.7 To change or stop the element file that has already started: Click on Analyze. A dialogue box will appear with these options:

Stop immediately

Stop after current replicate

Stop after completing all replicates for current sample

12.2.2.8 To restart the analysis from the beginning: In the Automated Analysis Control window, click on RESET. Start the analysis as usual.

12.2.2.9 To continue the analysis where it was interrupted or continue with a specific sample: In the Automated analysis Control window, click on Analyze All or Analyze Samples. A dialog box appear, elect one of the options. Click on OK.

12.2.2.10 Performing a recalibration: You define the type and frequency of automatic recalibration on the Checks page of the Method.

Stop the analysis.

In the Automated Analysis Control window, click on Reset.

In the Automated Analysis Control window, click on Calibrate.

12.2.2.11 Analyzing One or More Selected Samples: IN the Automated Analysis Control window, click on Select Location – a dialog box appears. Select the Analyze Samples In Locations option. Enter the autosampler locations of the solutions to analyze in the test box: Enter individual locations or a range of locations. Use comas to separate the locations and ranges, e.g. 10-15,18,20,25-30. Click OK.

12.2.2.12 Resetting the Autosampler: In the Automated Analysis Control window, click on Reset. Analyze the sample; the system will begin with the first sample in the sample list on the Set Up page of the Automated Analysis Control window.

12.2.2.13 Raising or Lower Sample Probe: In the Automated Analysis Control window, click on Probe Up/Down. The sample probe moves up or down but remains at the sample autosampler locations.

12.2.2.14 Rinsing the Sampling Probe and Sampling Tube: In the Automated Analysis Control window, select Location – a dialog box appears. Select “Go to Wash.” Click on OK. The sampling probe moves to the wash location. In the FIAS Control window: Make sure the FIAS-valve is in the correct position to rinse the sample channel. Click on the Pump.

12.2.2.15 The sample pump feeds the rinse fluid from the wash container through the sample channel. Click on Pump again to stop it. Click on Move Probe Up/Down to raise the probe out of the wash container in the automated analysis Control window.

12.2.3 System Shutdown

12.2.3.1 Place inlets of the carrier and reagent lines in a container of DI water. Rinse the sample, carrier, and reagent tubing with DI water. In the FIAS Control window, click on Valve Fill/Inject a number of times while the pump is running. This ensures that the sample channel and the inside of the FIAS-valve are rinsed effectively.

12.2.3.2 After adequate time, remove, all the tubes from the rinse containers. Click on Move Probe Up/Down to raise the sampling probe.

12.2.3.3 Allow the pump to run until all the tubes and the gas/liquid separator are empty. Then click on the Pump button to stop the pump.

12.2.3.4 Make sure that the FIAS-valve is in the Fill position.

12.2.3.5 Swing all the pressure levers away from the pump tube magazines.

12.2.3.6 Exit the AA WinLab application.

12.2.3.7 Switch off the FIMS spectrometer.

13. QUALITY CONTROL

13.1 Calibration Blanks – Calibration blanks (ICB and CCB) may not contain concentrations in excess of the Pace Reporting Limit. If blank results are not acceptable, analysis of the batch should be halted. Corrective actions could include preparation of fresh calibration standards and blanks.

13.2 Calibration Checks- Calibration checks (ICV and CCV) must agree within 10% of the true value. If the results are unacceptable, re-calibrate the system and then re-analyze the check solution and samples.

13.3 Pace Reporting Limit Standard (CRDL) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.4 Laboratory Control Spike – A Laboratory Control Spike (LCS) must be prepared and analyzed with every sample batch or every 20 samples, whichever is more frequent. A Laboratory Control Spike Duplicate (LCSD) is performed if there is insufficient sample available for a MS/MSD or if requested by the client. The LCS and LCSD must meet criteria 85-115% recovery or all the samples prepared in the batch must be re-prepped and re-analyzed. The acceptance criteria for precision is 20% RPD. If insufficient sample remains to re-prep and re-analyze, the data qualifiers is given to all associated samples.

13.5 Method Blank – A Method Blank (MB) must be prepared and analyzed with every sample batch or every 20 samples, whichever is more frequent. The MB must not contain mercury at a concentration at or above the Pace Reporting Limit. Any samples digested with an unacceptable method blank must be re-prepped and analyzed unless the sample

concentrations are less than the reporting limit or more than 10 times the value found in the method blank. In those cases that MDL reporting is required, the MB must be evaluated to the MDL. For MDL reporting, an appropriate data qualifier is given to samples associated with +/-MB hits between the +/-MDL and +/-PRL where the sample results are less than 10 times the value found in the method blank. Please refer to the attachment section for the flagging charts.

13.6 Matrix Spike/ Matrix Spike Duplicate – A Matrix Spike/Matrix Spike Duplicate (MS/MSD) must be prepared and analyzed for each group of samples that are similar in matrix at a frequency of 5%. The sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD. If one or both spike recoveries are outside 85-115% recovery acceptance criteria, the parent sample is given an appropriate data qualifier. If the RPD is outside 20% RPD criteria the parent sample is given an appropriate data qualifier. Parent sample is chosen at random or assigned by the client.

13.7 All reported results must be within the range of the calibration curve. Dilute when results are greater than the high standard in the curve.

14. DATA ANALYSIS AND CALCULATIONS

14.1.1 **Water Samples** – Since initial sample aliquot and final digestion volumes are the same, the mercury analyzer data system will calculate the concentration directly. No further calculations are necessary unless the sample was diluted.

14.1.2 **Soil/Solid/Biota Samples** -

Final Result (mg/kg dry weight corrected) =

$$\frac{\text{Raw data result (µg/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{Sample weight (g)} * \text{Dry weight for soil/solid (decimal form)}}$$

Biota results are reported on an as is/wet weight basis. The dry weight correction in the formula is not applicable.

14.1.3 **Wipes**

Final Result (µg/wipe) =

$$\frac{\text{Raw data result (µg/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{1 wipe}}$$

14.1.4 **Accuracy**-

$$\text{Spike Percent Recovery} = \frac{\text{Spike Sample Result} - \text{Sample Result}}{\text{Spike Added}} \times 100$$

14.1.5 **Precision**-

$$\text{Relative Percent Difference} = \frac{\text{Spike Sample Result} - \text{Spike Sample Duplicate Result}}{(\text{Spike Sample Result} + \text{Spike Sample Duplicate Result})/2} \times 100$$

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

Table D: Data Assessment and Acceptance Criteria

Analytical Method ⇨ Calibration Measure ⇩	SW846 7470A SW846 7471A/B Frequency	Acceptance Criteria
Laboratory Control Spike and Laboratory Control Spike Duplicate	• One LCS per batch of samples, up to 20 environmental samples, whichever is more frequent. • A LCSD is required if MS/MSD is not performed or if requested by the client.	• 85 –115% with 20% RPD
Matrix Spike / Matrix Spike Duplicate (MS/MSD)	• One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.	• 85 –115% with 20% RPD
Method Blank	• One per batch of samples, up to 20 environmental samples, whichever is more frequent.	• Less than the RL (LOWEST STANDARD IN CURVE)
Initial Calibration	• Analyzed daily before samples • Minimum 5 standards plus a blank	• Correlation coefficient must be 0.995 or greater
Initial Calibration Verification (ICV)	• Analyzed after calibration .	• Recovery must be between 90 – 110%
Initial Calibration Blank (ICB)	• Analyzed after ICV, but before samples.	• Less than RL (LOWEST STANDARD IN CURVE)
CRDL	• Analyzed after Initial Calibration.	• 60-140%
Continuing Calibration Verification (CCV)	• Analyzed after every 10 samples .	• Recovery between 90 – 110%
Continuing Calibration Blank (CCB)	• After each CCV, but before samples.	• Project specific or • Less than RL (LOWEST STANDARD IN CURVE)

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

Table E: CORRECTIVE ACTION

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation: SW846 7470A, SW846 7471A/B If these conditions are not achieved ⇒**
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
Initial / Continuing Calibration Verification	• 5
Initial / Continuing Calibration Blank	• 6
Holding Time Compliance	• 7
CRDL	• 8

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 times the method blank contamination are reported without qualification. For MB detections greater than the reporting limit, correct the problem and re-digest the samples provided there is sufficient sample amount. If the is insufficient sample amount, qualify the data, generate a Non-Conformance Memo, and talk with the project manager to determine further action.

2. If the MS and MSD fails precision or accuracy control limits flag the parent with the appropriate data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifier to reflect the deficiencies. Generate a Non-Conformance Memo.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. If ICV/CCV is outside the control limits, reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed.

6. If ICB/CCB is outside the control limits, reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed unless the sample concentration is $\geq$10X the blank detection or the sample is an ND for blank failures that have a + bias.

7. Notify Project Manager by submitting a LabTrack Ticket and flag with the appropriate data qualifier.

8. If CRDL is outside the control limits reanalyze the CRDL to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. All applicable samples must be reanalyzed.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 See Table E: CORRECTIVE ACTION

18. METHOD PERFORMANCE

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

18.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of LOD and LOQ*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

18.1.4 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

19. METHOD MODIFICATIONS

19.1 The method has been modified to perform digestion and analysis on biological tissue samples.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

20.1 See PE FIMS 100 operator's manual for information.

21. TROUBLESHOOTING

21.1 See PE FIMS 100 operator's manual for information.

22. SAFETY

22.1 Standards and Reagents - The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses.

22.2 Material Safety Data Sheets (MSDSs) – A reference file of MSDS are on file in the laboratory and available to all personnel. A formal safety plan has been prepared and distributed to all personnel with documented training.

22.3 Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion. All digestions must be conducted in a properly functioning fume hood.

22.4 Samples - Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.

22.4.1 Extreme caution must be used when preparing rodents for digestion. The samples must undergo a special procedure to destroy any Hantavirus, which may be present. Refer to the most recent version of SOP S-GB-L-002 *Small Rodent Handling and Homogenization* for details.

22.5 Analysts should take necessary safety precautions when handling chemicals and samples. Proper personal protective equipment may include safety gloves, lab coats, and safety glasses or goggles. Analysts should be familiar with the MSDS sheets for all chemicals and reagents they use for this procedure and the location of the MSDS sheets within the laboratory. Any questions or concerns should be taken to the laboratory Chemical Hygiene/Safety Officer.

23. WASTE MANAGEMENT

23.1 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Handling and Management*.

24. POLLUTION PREVENTION

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. REFERENCES

25.1 SW-846 7470A, Revision 1, September 1994

25.2 SW-846 7471A, Revision 1, September 1994

25.3 SW-846 7471B, Revision 2, February 2007

25.4 PASI Quality Manual, current revision

25.5 National Environmental Laboratory Accreditation Conference (NELAC), July 2003 Standards

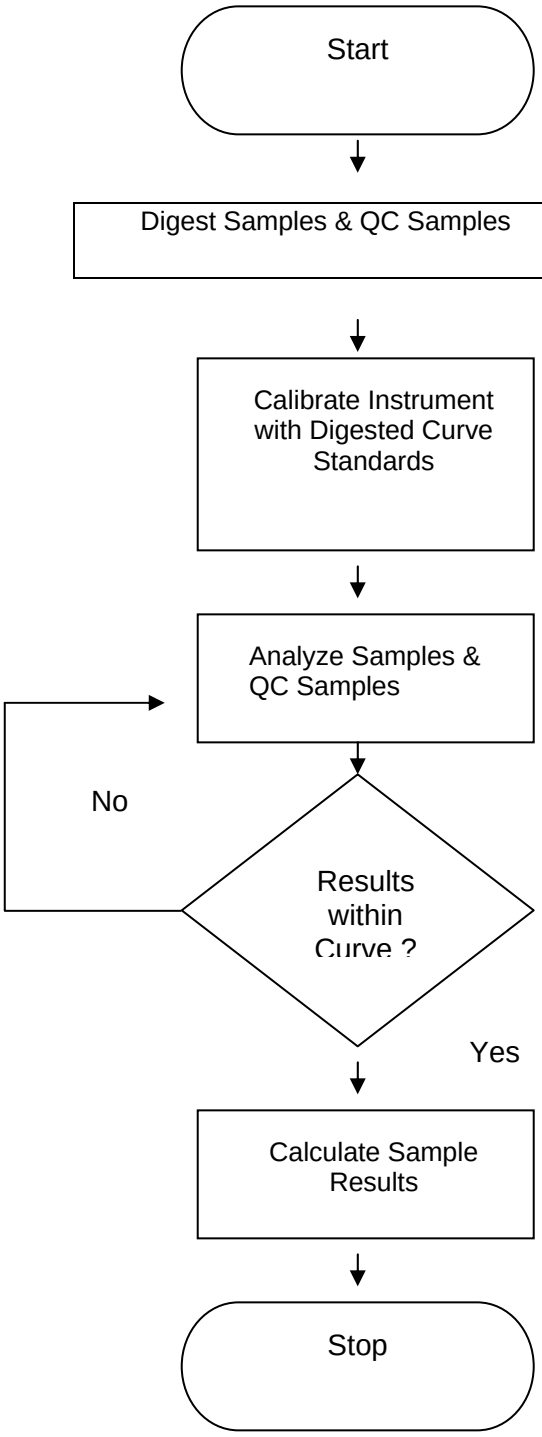
26. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ETC.

26.1 Mercury Process Flow Chart

27. REVISIONS

Document Number	Reason for Change	Date
GB-I-036-Rev.0	Combined G2-MET-17 Rev.2 and G2-MET-Rev1 into 1 SOP in new format.	August 28, 2006
GB-I-036-Rev.1	Added Reference to Wipes	December 15, 2006
GB-I-036-Rev.2	Updated Signature Page Updated Section 4.4 - Samples may be diluted to decrease the chlorides Added Section 6.2 Updated Tables 2 and 3. Added Sections 12.1.1.11 and 12.1.1.12 Added Sections 12.1.2.4 and 12.1.2.5 Added Sections 12.2.1, 12.2.3 thru 12.2.10 Updated Section 14	January 2, 2008
S-GB-M-004-REV.00	Updated Method Reference from SW846 7471A to SW846 7471B throughout document. Updated SOP reference to S-GB-M-004-REV.00. Section 4.4 – Added last sentence as alternative Section 5.4.1 – Update SOP reference and name Section 6.2 – Changed biota control blank source from “must” to “should” be farm raised. Section 7 – Deleted Renumbered document Section 10.4 – Added PRLS Table 3 – Added PRLS to HgCal 2 standard Section 12.3 – Added PRLS Section 12.4, 12.5, and 12.6 – Updated flagging language to reflect EpicPro. Section 12.5 – Changed MB contamination from 20 times to 10 times to reflect 7471B requirements. Section 15 – Updated SW-846 7471 reference Flagging Charts A, B, and C – Where ever “A flag” appeared, changed to “appropriately flag”. Flagging Charts A, B, and C – Where ever “20 times” appeared, changed to “10 times”.	September 12, 2008
S-GB-M-004-REV.01	Updated Signature Page	May 26, 2009
S-GB-M-004-Rev.02	Updated signature page Section 11.1.1.15 – Updated Solution Table 1 – Updated Vendors Tables 2 and 3 – Changed Hg CCV Standard to Hg Cal Intermediate	12Apr2010
S-GB-M-004-Rev.03	Updated Signature Page Added Method 7471A to title Section 15: Updated References (7471A)	14Sept2010
S-GB-M-004-Rev.04	Updated SOP references throughout Document. Updated format following current revision of SOP: S-GB-Q-017 *Preparation of SOPs.* Attachment I, II, III: Removed MDL/RL reporting charts and incorporated information into body of Section 13.	07Dec2012

Mercury Process Flow Chart



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STANDARD OPERATING PROCEDURE

DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) SPECTROSCOPY BY 6010B

REFERENCE METHODS: SW-846 METHOD 6010B WITH 3010A AND 3050B

LOCAL SOP NUMBER:	S-GB-M-014-Rev.03
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APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/19/12

Kate Grams, Laboratory Quality Manager — Date: 12/7/2012

Chad Rusch, Department Manager — Date: 12/07/2012

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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17. REVISIONS 30

1. PURPOSE

This Standard Operating Procedure (SOP) documents the procedures used by PASI – Wisconsin to determine the concentration of specific metals in environmental water, wipe, paint chips, sludges, soils, and solid samples. The laboratory utilizes the ICP (Inductively Coupled Plasma – Optical Emission Spectrometer) and bases these documented procedures on those listed in EPA SW-846 Method 6010B. Sample preparation procedures are based on SW-846 Methods 3010A and 3050B.

2. SUMMARY OF METHOD

2.1 Samples are digested, excluding filtered groundwater, by heating with appropriate acids and oxidizing agents to solubilize the target elements. Portions of the digestates (or filtered, acidified groundwater samples) are pumped into a nebulizer to produce an aerosol. The aerosol is aspirated into the torch of Inductively Coupled argon Plasma Optical Emission Spectrometer (ICP-OES) where it is evaporated and decomposed into atoms and ions. The plasma energy causes the target atoms to become excited and, during relaxation, emit characteristic light in the visible and/or ultraviolet emissions. Each element in the sample emits photons at a discrete wavelength(s), which are specific to that element. The light emissions are separated into wavelength and order by passing through a prism and onto an Echelle grating. The signal is then read and quantified by a Charge Injection Device (CID). The intensities of the wavelengths are proportional to the quantity of the target elements that is determined through a comparison to a know concentration (a calibration curve). The signals received from the CID are digitized and relayed to the instrument computer as an analytical signal.

2.2 Background correction may be required to compensate for spectral interferences. Background is measured adjacent to analyte lines at a wavelength selected to be free of spectral interference and which reflects the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a correction would actually degrade the analytical result.

3. SCOPE AND APPLICATION

3.1 This procedure may be used to determine concentrations of trace metals in water, wipe, paint chips, sludges, soils, and solid samples. A list of applicable compounds and Practical Reporting Limits (PRL) for aqueous, solid and TCLP samples are shown in Table 3.1.

Table 3.1 – Elements and associated PRLs for water and leachates

	Element	Wavelength (nm)	PRL Aqueous (µg/L)	PRL Solid* (mg/kg)	PRL TCLP/SPLP/ASTM (mg/L)
Ag	Silver	328.0	10.0	1.0	0.250
Al	Aluminum	396.1	500	50.0	2.50
As	Arsenic	189.0	20	2.0	0.250
B	Boron	208.9	100	10.0	0.500
Ba	Barium	455.4	5.0	0.50	2.50
Be	Beryllium	313.0	4.0	0.40	0.250
Ca	Calcium	317.9	200	20	5.000
Cd	Cadmium	228.8	5.0	0.50	0.005
Co	Cobalt	228.6	5.0	0.50	0.250
Cr	Chromium	267.7	5.0	0.50	0.250
Cu	Copper	324.7	10	1.0	0.250
Fe	Iron	259.9	100	10.0	2.50
K	Potassium	766.4	200	20.0	5.000
Mg	Magnesium	279.0	200	20.0	5.000
Mn	Manganese	257.6	5.0	0.50	0.250
Mo	Molybdenum	202.0	20	2.0	0.250
Na	Sodium	589.5	1000	100.0	5.000
Ni	Nickel	231.6	10	1.0	0.250
Pb	Lead	220.3	7.5	1.0	0.0075
Sb	Antimony	206.8	20	2.0	0.250
Se	Selenium	196.0	20	2.0	0.250
Sn	Tin	189.9	50	5.0	0.250
Sr	Strontium	407.7	5.0	0.50	0.250
Ti	Titanium	334.9	5.0	0.50	0.250
Tl	Thallium	190.8	40	4.0	0.250
V	Vanadium	292.4	5.0	0.50	0.250
Zn	Zinc	206.2	40	4.0	0.250

* Based on standard weights/volumes (0.5g/50mL). Final RLs for individual samples may vary depending in actual weights/volumes and percent solids used. Soil include soils, sludges, paint chips, wipes, and solids. (Unit for wipes is in Total µg.)

Soils are reported on a dry weight basis.

3.2 This procedure is restricted to use by, or under the supervision of, analysts experienced in the digestion of samples for metals analysis and analysis of digestates by ICP. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.3 This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

3.4 Current Method Detection Limits can be found in current LIMS or can be provided by the Quality Department.

4. INTERFERENCES

4.1 Spectral Interferences – Overlap of emission lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light can contribute to spectral interferences. These interferences can typically be minimized by careful selection of quantitation wavelengths, interelement corrections, and background correction.

4.2 Physical Interferences – Changes in sample viscosity, surface tension, or other effects associated with sample transport and nebulization can produce significant inaccuracies, especially in samples containing high concentrations of dissolved solids and acids. Dissolved solids may build up on the nebulizer tip, altering the sample flow rate and causing instrument drift. These effects can be minimized by sample dilution or use of a specially designed high-solids nebulizer. Use of internal standards helps in recognizing sample introduction issues.

4.3 Chemical Interferences – Molecular compound formation, ionization effects, and solute vaporization effects are typically not significant with ICP determinations. If observed, they can be minimized by careful selection of plasma and spectrometer operating parameters.

4.4 Memory Interferences – Sample deposition on the nebulizer tubing, spray chamber, and plasma torch can cause apparent sample carryover. Memory interferences can be minimized by flushing the system with rinse blanks between samples. If memory interference is suspected for a sample, the sample must be re-analyzed after a sufficient rinse period.

4.5 High Salt Concentrations – High salt concentrations in sample digestates can cause signal suppression and confuse interference tests. Use of internal standards helps in recognizing signal suppression.

5. SAFETY

5.1 Standards and Reagents

5.2 The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound shall be treated as a potential health hazard. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Special care shall be taken when handling the high-concentration acids and oxidizing reagents used for sample digestion. All digestions must be conducted in a properly functioning fume hood. The use of personal protective equipment (gloves, lab coats and safety glasses) is required.

5.3 Samples

5.3.1 Although sample check-in should be notified of any hazardous samples, samples shall always be considered as “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.

5.4 Equipment

5.4.1 ICP Radio Frequency (RF) Generator – The RF generator used to induce the atomic plasma produces a high energy radio emission. The electrical requirements for this equipment are substantial. The RF generator must only be serviced by those trained specifically for service and repair of the instrument.

5.4.2 ICP RF Coil – The wound coil around the top of the torch produces a high energy, oscillating radio frequency field. The field is substantial and may interfere with surrounding electronics including implanted medical devices. Those individuals with such devices (i.e. pacemakers) must stay clear of this instrument while in operation.

6. DEFINITIONS

6.1 Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions. In addition to those listed in the QAM, the following are additional terms found in this SOP.

6.2 Instrumental Detection Limit (IDL) – The concentration equivalent to a signal, due to the analyte, which is equal to the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day at the same wavelength.

6.3 Instrument Check Standard – A multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. This is the same as the Continuing Calibration Verification Standard (CCV) referred to in the calibration section of this SOP.

6.4 Spectral Interference Check Solution (SIC) – A solution of selected analytes of higher concentrations used to evaluate the procedural routine for correcting known inter-element spectral interferences. This is also known as the Inter-element Correction Solution (ICSA).

6.5 Linear Detection Range – Defined as the upper limit of quantitation for an analyte. The LDR is determined as the upper range limit of an observed signal that is no more than 10% below the level extrapolated from the lower standards.

6.6 Interference Check Sample (ICSAB) – A solution containing both interfering and analyte elements of known concentration that can be used to verify background and inter-element correction factors.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Collection, Preservation, Storage and Hold Times

Sample type	Collection per sample	Preservation	Storage	Hold time
Soil/ Solid/ Wipe/Paint Chip/Sludge	Pre-cleaned plastic or glass containers. Refer to Pace S-ALL-Q-021, Sub-Sampling SOP, for obtaining representative aliquots.	No Preservation	Refrigerated at 4+/- 2°C	Up to 6 months
Total Metals –Aqueous	Pre-cleaned plastic containers with HNO_3	Nitric Acid (HNO_3) to pH<2, preserved at time of collection	Room Temperature	Up to 6 months
Dissolved Metals – Aqueous(1)	Pre-cleaned plastic containers – Filter sample at time of collection through a 0.45µm membrane filter.	Nitric Acid (HNO_3) to pH<2, filtered then preserved at time of collection.	Room Temperature	Up to 6 months
TCLP/SPLP/ASTM(1)	Pre-cleaned plastic containers	-Filtered then preserved at time of digestion. -Refrigerate at 4±2 °C until preservation. -Nitric Acid (HNO_3) to pH<2	Refrigerate at 4±2 °C	From Field Collection to Leach Extraction: 180 Days. From Digestion to Determinative Analysis: 180 Days. (Total Elapsed Time: 360 Days).

(1) If filtration cannot be performed in the field, the sample must be taken in a pre-cleaned, unpreserved plastic container and transported to the lab as soon as possible. On arrival at the lab, the sample must be filtered and then preserved to a pH <2. The sample must sit for **24 hours** prior to preparation. This is to ensure that the sample does not have a buffering effect that raises the pH to above <2. The data is qualified if sample preservation fails to bring pH down to ≤2. (Note: TCLP Extracts are not required to sit for 24 hours prior to digestion after the addition of nitric acid.)

7.2 Preservative Check

The pH of samples must be verified to be pH<2 and documented in the Sample Receiving or Metals pH logbook prior to aliquoting for analysis. If a sample has a pH>2, additional preservative must be added to return the sample to <2. Acid volume shall not to exceed 2% of the container capacity. Once adjusted, the sample must be allowed to sit for 24 hours prior to preparation. If the sample is not able to maintain a preservation of a pH of<2, then it must be qualified as such.

8. EQUIPMENT AND SUPPLIES

8.1 Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
ICAP System	Thermo Scientific	ICAP 6500	Serial Number: 20073913
Autosampler	Cetac	ASX-520	Serial Number: 080735A 520
Data System	iTEVA	2.0.039	
Recirculator	Neslab	ThermoFlex900	Serial Number: 107271037
Analytical Balance			

8.2 General Supplies

Item	Vendor (or equivalent)	Model / ID (or equivalent)	Catalog # (or equivalent)	Description
Argon – high purity grade	PraxAir			Bulk
Autosampler Vials	Fisher Scientific	15 mL Polypropylene Test Tubes	14-375-150	
Autosampler Pump Tubing	Analytical West		PT-2180PS	red/red 1.14mm ID – Carrier
			PT-2200SAS	1.30mm ID – Waste
			PT-2080PS-F	orange/orange 0.38mm ID – Internal Standard
Digestion Vials	Environmental Express		SC499	Pre-cleaned polypropylene 70-mL screw cap vials.
Watch Glass	Environmental Express			Polypropylene, to fit 70-mL digestion vial
Heating Block	Environmental Express			Polypropylene, to fit 70-mL digestion vial
Mechanical Pipettes and Tips	Eppendorf			Or equivalent
Wash Bottle				
Filter paper	Whatman		541 Filter Paper	
pH Strips				
Y Connectors	Hewlett-Packard		G1820-65106	
Duo Torch / Duo Ceramic Cone	Thermo Fisher Scientific		8423-120-51241 / 84223-120-51261	
Spray Chamber/Spray Chamber adapter	Thermo Fisher Scientific		8423-120-51411 / 8423-120-51251	
2mm Center Tube	Thermo Fisher Scientific		8423-120-51371	
SC-FAST Probe	ESI		SC-5037-3995-150	7” Teflon/Carbon Probe 1.0mmID
SC-FAST Waste Line	ESI		SC-0323-0002-215	Waste Line
SC-FAST Valve	ESI	F7	SC-0599-1024	F7 Valve Head
	ESI		ES-2501-PPF2	
	ESI		ES-2501-PPMZ	
Peri-Pump	ESI			Fluoropolymer union, barbed
Nebulizer Line	ESI		SC-0317-0250-30	
Nuts and Ferrules	ESI		SC-0599-0116-K	For high flow 1/16” tubing
Nuts and Ferrules	ESI		SC-0599-0108-W	For high flow 1/8” tubing
Vacuum Line	ESI		SC-0321-32-215	For SC-FAST high flow valve
Sample Loop	ESI		SC-0318-15	1.5mL sample loop
Nebulizer	ESI		ES-2040	Micro Flow PFA-St nebulizer

8.3 Glassware

Glassware	Vendor / Item # / Description
Volumetric Flasks	Class A
Graduated Cylinders	Class A

9. REAGENTS AND STANDARDS

9.1 Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
De-ionized Nano-pure water (DI)	ASTM Type II, or equivalent (18mOhm or higher resistivity)	Nanopure Water
Nitric Acid (HNO_3)	Trace metals grade, Fisher Insta-analyzed, or equivalent	J.T Baker Cat #9598-34 or equivalent
Hydrochloric Acid (HCl)	Trace metals grade, Fisher Insta-analyzed, or equivalent	Fisher Cat # A508 or equivalent
Hydrogen Peroxide (H_2O_2)	30% certified reagent grade; trace metals grade if available. Stabilized solutions come with Sodium added.	Fisher Cat # H325-4 or equivalent
Acidified Nano-pure water	5mL Nitric Acid and 5mL Hydrochloric Acid per 100mL Nano-pure water	

9.2 Standards Definitions, Storage, and Sources

9.2.1 Definitions

Standard	Description	Comments
Initial Calibration Standard(s)	Standards prepared from single and/or multi-element standard(s) at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range for single point calibration.	Calibration standards can be made and used for 6 months unless a standard used to make them expires earlier.
Initial Calibration Verification Standard	Standard prepared near the midpoint of the calibration range and is used to verify the accuracy of the calibration.	Must be prepared from a source independent of CCV and Calibration Solutions.
Continuing Calibration Verification Standard	Standards prepared from single and/or multi-element standards at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range. This standard verifies the accuracy of the calibration curve.	May be prepared from the same source as the calibrations standards
Low Level Check Standard (CRDL)	Must be performed if the lowest non-zero calibration point is not at or below the reporting limit. The recovery must be between 50 and 150% to report sample data. The recovery must be between 60 and 140% for samples reported to the State of MN.	NELAC requirement for single point calibrations, a client specific requirement for certain QAPPs
Inter-element correction solution (ICSA)	A solution prepared with high concentrations of interfering elements (Al, Ca, Fe, Mg, …). This is run daily at a minimum to verify the inter-element correction factors.	Al, Ca, Fe, Mg
Inter-element correction solution (ICSAB)	A solution prepared with high concentrations of interfering elements and low level concentrations of the other target elements. This is analyzed by the Green Bay Laboratory per client request, and for samples requiring QC Level 3 and 4 data packages, immediately following the ICSA solution.	Not a 6010B requirement but may be required on a client specific basis.
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard solution contains a non-target element (yttrium (Y)) at a concentration yielding an appropriate final concentration in the sample.	Y at 5ppm with Li. Added in-line to all samples and standards.
Single Element Standards	Stock standards purchased from venders containing one element. Used for checking IECs and may be used for checking linear ranges.	Must be 99.99% pure.
Spiking Standard	This solution contains all target analytes and should not be prepared from the same standards as the calibration standards.	

9.2.2 Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	▪ Concentrated reference solution purchased directly from approved vendor	▪ Manufacturer's recommended expiration date for unopened standards.	▪ Manufacturer's recommended storage conditions ▪ When standard is opened, record all information in the standard logbook.
Intermediate and Working Standard Solutions	▪ Reference solutions prepared by dilutions of the stock solution	▪ Should be prepared as needed or at least every 6 months. The intermediate or working standard can not exceed the expiration date recommended by the manufacturer for each component used.	▪ Store at room temperature

9.3 Standard Sources

9.3.1 Standards are prepared from commercially available multi-compound stock solutions. The sources of the stock solutions, formulas for preparing dilutions and working standards, and concentrations for all compounds are presented in tables within section 9.3. All intermediate and working standards are prepared using acidified Nano-pure water and used immediately or stored for no longer than 6 months.

9.3.2 Analytical Working Standard Preparations - Depending on the volume of each solution needed, the standards are brought to volume in volumetric flasks with Nano-pure water unless otherwise noted.

Table 9.3.1 – Calibration Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
Spex #1 (Spex MixStd 1-100)	As	100 ppm	2 ml	10 mL HNO3/ 10 mL HCl	200 ml	1.0 ppm
	Ba	100 ppm				1.0 ppm
	Be	100 ppm				1.0 ppm
	Cd	100 ppm				1.0 ppm
	Co	100 ppm				1.0 ppm
	Cr	100 ppm				1.0 ppm
	Cu	100 ppm				1.0 ppm
	Mn	100 ppm				1.0 ppm
	Ni	100 ppm				1.0 ppm
	Pb	100 ppm				1.0 ppm
	Se	100 ppm				1.0 ppm
	Sr	100 ppm				1.0 ppm
	Tl	100 ppm				1.0 ppm
	V	100 ppm				1.0 ppm
	Zn	100 ppm				1.0 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	4 ml			2.0 ppm
	Mo	100 ppm				2.0 ppm
	Sb	100 ppm				2.0 ppm
	Sn	100 ppm				2.0 ppm
	Ti	100 ppm				2.0 ppm
	Ag	50 ppm				1.0 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	10 ml			50 ppm
	Ca	1000 ppm				50 ppm
	Fe	1000 ppm				50 ppm
	K	1000 ppm				50 ppm
	Mg	1000 ppm				50 ppm
	Na	1000 ppm				50 ppm

Table 9.3.2 – CCV Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
Spex #1 (Spex MixStd 1-100)	As	100 ppm	5.0 ml	50 mL HNO3/ 50 mL HCl	1000 ml	0.5 ppm
	Ba	100 ppm				0.5 ppm
	Be	100 ppm				0.5 ppm
	Cd	100 ppm				0.5 ppm
	Co	100 ppm				0.5 ppm
	Cr	100 ppm				0.5 ppm
	Cu	100 ppm				0.5 ppm
	Mn	100 ppm				0.5 ppm
	Ni	100 ppm				0.5 ppm
	Pb	100 ppm				0.5 ppm
	Se	100 ppm				0.5 ppm
	Sr	100 ppm				0.5 ppm
	Tl	100 ppm				0.5 ppm
	V	100 ppm				0.5 ppm
	Zn	100 ppm				0.5 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	10.0 ml			1.0 ppm
	Mo	100 ppm				1.0 ppm
	Sb	100 ppm				1.0 ppm
	Sn	100 ppm				1.0 ppm
	Ti	100 ppm				1.0 ppm
	Ag	50 ppm				0.5 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	25.0 ml			25 ppm
	Ca	1000 ppm				25 ppm
	Fe	1000 ppm				25 ppm
	K	1000 ppm				25 ppm
	Mg	1000 ppm				25 ppm
	Na	1000 ppm				25 ppm

Table 9.3.3 – ICV (Second Source) Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
IV #1 (PA-STD-1B)	As	200 ppm	0.5 ml	10 mL HNO3/ 10 mL HCL	200 ml	0.5 ppm
	Ba	200 ppm				0.5 ppm
	Be	200 ppm				0.5 ppm
	Cd	200 ppm				0.5 ppm
	Co	200 ppm				0.5 ppm
	Cr	200 ppm				0.5 ppm
	Cu	200 ppm				0.5 ppm
	Mn	200 ppm				0.5 ppm
	Ni	200 ppm				0.5 ppm
	Pb	200 ppm				0.5 ppm
	Se	200 ppm				0.5 ppm
	Sr	200 ppm				0.5 ppm
	Tl	200 ppm				0.5 ppm
	V	200 ppm				0.5 ppm
	Zn	200 ppm				0.5 ppm
IV #2 (PA-STD-2B	B	200 ppm	1.0 ml			1.0 ppm
	Mo	200 ppm				1.0 ppm
	Sb	200 ppm				1.0 ppm
	Sn	200 ppm				1.0 ppm
	Ti	200 ppm				1.0 ppm
	Ag	100 ppm				0.5 ppm
IV #3 (PA-STD-3B)	Al	2000 ppm	2.5 ml			25 ppm
	Ca	2000 ppm				25 ppm
	Fe	2000 ppm				25 ppm
	K	2000 ppm				25 ppm
	Mg	2000 ppm				25 ppm
	Na	2000 ppm				25 ppm

Table 9.3.4 – Spiking Standard (for LCS and MS/MSD) Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)	Spike Concentration*
IV #1 (PA-STD-1B)	As	200 ppm	25 ml	12 mL HNO3	200 ml	50 ppm	500 ppb
	Ba	200 ppm				50 ppm	500 ppb
	Be	200 ppm				50 ppm	500 ppb
	Cd	200 ppm				50 ppm	500 ppb
	Co	200 ppm				50 ppm	500 ppb
	Cr	200 ppm				50 ppm	500 ppb
	Cu	200 ppm				50 ppm	500 ppb
	Mn	200 ppm				50 ppm	500 ppb
	Ni	200 ppm				50 ppm	500 ppb
	Pb	200 ppm				50 ppm	500 ppb
	Se	200 ppm				50 ppm	500 ppb
	Sr	200 ppm				50 ppm	500 ppb
	Tl	200 ppm				50 ppm	500 ppb
	V	200 ppm				50 ppm	500 ppb
	Zn	200 ppm				50 ppm	500 ppb
IV #2 (PA-STD-2B)	B	200 ppm	25 ml			50 ppm	500 ppb
	Mo	200 ppm				50 ppm	500 ppb
	Sb	200 ppm				50 ppm	500 ppb
	Sn	200 ppm				50 ppm	500 ppb
	Ti	200 ppm				50 ppm	500 ppb
	Ag	100 ppm				25 ppm	250 ppb
IV #3 (PA-STD-3B)	Al	2000 ppm	25 ml			500 ppm	5000 ppb
	Ca	2000 ppm				500 ppm	5000 ppb
	Fe	2000 ppm				500 ppm	5000 ppb
	K	2000 ppm				500 ppm	5000 ppb
	Mg	2000 ppm				500 ppm	5000 ppb
	Na	2000 ppm				500 ppm	5000 ppb

*** Use 1.0 ml of spiking std to 50 ml**

Table 9.3.5 – Low Level Check Standard (CRDL) Intermediate

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)*	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppb)
4400-100118CS01	Ag	10 ppm	10 ml	5 mL HNO3/ 5 mL HCl	100 ml	1,000 ppb
	Al	500 ppm				50,000 ppb
	As	20 ppm				2,000 ppb
	B	100 ppm				10,000 ppb
	Ba	5 ppm				500 ppb
	Be	4 ppm				400 ppb
	Ca	200 ppm				20,000 ppb
	Cd	5 ppm				500 ppb
	Co	5 ppm				500 ppb
	Cr	5 ppm				500 ppb
	Cu	10 ppm				1,000 ppb
	Fe	100 ppm				10,000 ppb
	K	1000 ppm				100,000 ppb
	Mg	200 ppm				20,000 ppb
	Mn	5 ppm				500 ppb
	Mo	20 ppm				2,000 ppb
	Na	1000 ppm				100,000 ppb
	Ni	10 ppm				1,000 ppb
	Pb	7.5 ppm				750 ppb
	Sb	20 ppm				2,000 ppb
	Se	20 ppm				2,000 ppb
	Sn	50 ppm				5,000 ppb
	Sr	5 ppm				500 ppb
	Ti	5 ppm				500 ppb
	Tl	40 ppm				4,000 ppb
	V	5 ppm				500 ppb
	Zn	40 ppm				4,000 ppb

Table 9.3.5b – Low Level Check Standard (CRDL)

Low Level Check Standard Intermediate	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)*	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppb)
CRDL Intermediate (In Table 9.3.5)	Ag	1,000 ppb	2 ml	10 mL HNO3/ 10 mL HCl	200 ml	10 ppb
	Al	50,000 ppb				500 ppb
	As	2,000 ppb				20 ppb
	B	10,000 ppb				100 ppb
	Ba	500 ppb				5 ppb
	Be	400 ppb				4 ppb
	Ca	20,000 ppb				200 ppb
	Cd	500 ppb				5 ppb
	Co	500 ppb				5 ppb
	Cr	500 ppb				5 ppb
	Cu	1,000 ppb				10 ppb
	Fe	10,000 ppb				100 ppb
	K	100,000 ppb				1000 ppb
	Mg	20,000 ppb				200 ppb
	Mn	500 ppb				5 ppb
	Mo	2,000 ppb				20 ppb
	Na	100,000 ppb				1000 ppb
	Ni	1,000 ppb				10 ppb
	Pb	750 ppb				7.5 ppb
	Sb	2,000 ppb				20 ppb
	Se	2,000 ppb				20 ppb
	Sn	5,000 ppb				50 ppb
	Sr	500 ppb				5 ppb
	Ti	500 ppb				5 ppb
	Tl	4,000 ppb				40 ppb
	V	500 ppb				5 ppb
	Zn	4,000 ppb				40 ppb

Table 9.3.6– Inter-element Correction Solution (ICSA) Working Dilutions and Concentrations

Pace Standard #	Vendor	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (mg/L)	Stock Concentration (µg/mL)
Single Element Al	Ultra Scientific	ICP-113	10	200	Al	500	10000
Single Element Ca	Ultra Scientific	ICP-120	10	200	Ca	500	10000
Single Element Mg	Ultra Scientific	ICP-112	10	200	Mg	500	10000
Single Element Fe	Ultra Scientific	ICP-126	4	200	Fe	200	10000

Table 9.3.6b– Inter-element Correction Solution (ICSAB) Working Dilutions and Concentrations – Include as needed.

Pace Standard #	Vendor	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (µg/mL)	Stock Concentration (µg/mL)
Single Element Al	Ultra Scientific	ICP-113	9.5	200	Al	475	10000
Single Element Ca	Ultra Scientific	ICP-120	9.5	200	Ca	475	10000
Single Element Mg	Ultra Scientific	ICP-112	9.5	200	Mg	475	10000
Single Element Fe	Ultra Scientific	ICP-126	3.5	200	Fe	175	10000
IV #1 (PA-STD-1B)	Inorganic Ventures	PA-STD-1B	0.5	200	As	0.5	200 ppm
					Ba	0.5	200 ppm
					Be	0.5	200 ppm
					Cd	0.5	200 ppm
					Co	0.5	200 ppm
					Cr	0.5	200 ppm
					Cu	0.5	200 ppm
					Mn	0.5	200 ppm
					Ni	0.5	200 ppm
					Pb	0.5	200 ppm
					Se	0.5	200 ppm
					Sr	0.5	200 ppm
					Tl	0.5	200 ppm
					V	0.5	200 ppm
					Zn	0.5	200 ppm
IV #2 (PA-STD-2B)	Inorganic Ventures	PA-STD-2B	1.0	200	B	1.0	200 ppm
					Mo	1.0	200 ppm
					Sb	1.0	200 ppm
					Sn	1.0	200 ppm
					Ti	1.0	200 ppm
					Ag	0.5	100 ppm
IV #3 (PA-STD-2B)	Inorganic Ventures	PA-STD-3B	2.5	200	Al	25	2000 ppm
					Ca	25	2000 ppm
					Fe	25	2000 ppm
					K	25	2000 ppm
					Mg	25	2000 ppm
					Na	25	2000 ppm
Single Element Ni	Ultra Scientific	ICP-028	0.1	200	Ni	1	1000 ppm

Table 9.3.7– Internal Standard Solution Working Dilutions and Concentrations

Pace Standard #	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (mg/L)	Stock Concentration (mg/L)
IS	ICP69N-1 (Accustandard)	5	1000	Yttrium	5 mg/L	1000
	ICP-103-5 (Ultra Scientific)	100	1000	Lithium	1000 mg/L	10000

10. CALIBRATION

10.1 Profile

The iCAP 6500 ICP will automatically check a carbon reference line against ambient air conditions each time the plasma is ignited to maintain wavelength accuracy.

10.2 Daily Initial Calibration

Allow the instrument 30 minutes to warm up after igniting the torch. The ICP must be calibrated each time it is set up for analysis or every 24 hours according to the manufacturer's instructions. Calibration requires analysis of a Calibration Blank and at least one level of calibration solution. Calibration standards can be made and used for 6 months unless a standard used to make them expires earlier. It would then expire on that date. Instrument standardization date and time must be recorded in the raw data. See Table 9.3.1 for details on the preparation of the Initial Calibration Standard.

10.3 Calibration Verification

10.3.1 Initial Calibration Verification (ICV) / Second Source

To assess the accuracy relative to the purity of the calibration standards, a standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as **Secondary Source Verification** or, alternatively as **Initial Calibration Verification**. This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. See the most current version of SOP S-GB-Q-026 *Standard and Reagent Management and Traceability* for more information of standards. This standard must be analyzed immediately after the calibration and before any batch QC or client samples. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$$\% \text{ Difference} = [\text{Result}_{ICV} - \text{TrueValue}_{ICV}] / \text{TrueValue}_{ICV} * 100$$

See Table 9.3.3 for details on the preparation of this standard. See Table 10.2 for control criteria

10.3.2 Reporting Limit Verification Standard (CRDL)

With every Initial Calibration, a standard corresponding to the practical reporting limit (PRL) must also be analyzed and meet established acceptance criteria.

Note: If a multi-point calibration is being run on the ICP, results detected below the lowest level calibration standard analyzed can only be reported if a PRL standard is also run and verified with that ICAL. If a PRL standard is not analyzed with that ICAL, results detected below the lowest calibration standard run for the multi-point calibration may not be reported. If the lowest calibration point corresponds to the PRL, this requirement is not applicable. The limits for the CRDL are +/- 50% of the true concentration, for work from MN the limits are +/-40%. See Table 9.3.5 for details on the preparation of this standard.

10.3.3 ICSA (Interference Check Standard) Verification

With every Initial Calibration, the inter-element check solution standard must be run to verify the inter-element correction factors. At a minimum, the ICSA standard must be analyzed at the beginning of the run, following the initial calibration and evaluated against the criteria in Table 10.2. If client specific requirements dictate that the ICSA must be analyzed at both the beginning and end of the analysis, the ICSA may be analyzed immediately after the completion of those specific samples and is not required to be placed at the very end of the sequence. If client specific requirements dictate that the ICSAB be run, the ICSAB must be analyzed immediately following the ICSA and be held to the criteria in Table 10.2.

10.3.4 Continuing Calibration Verification (CCV)

As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as **Continuing Calibration Verification**. The validity of the initial calibration is checked after every 10 samples and at the end of an analytical sequence by analyzing a midpoint calibration standard (CCV).

The values obtained from the analysis of the CCV are compared to the true values and a percent difference calculated according to the formula in Section 10.2.1. The percent difference must meet the method specified criteria in Table 10.2 for the analysis to proceed.

Table 10.2 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Linear Regression for elements requiring multiple calibration levels	Correlation coefficient ≥ 0.995	If not met, remake standards and recalibrate. One point calibration is standard for the Wisconsin laboratory.
Initial Calibration Verification Standard (Second Source)	Immediately after each initial calibration	≤±10% of true value The % RSD between replicate integrations of an ICV std must be <5% (min of 2 integrations).	If 4 or more replicates are used for analysis the RSD must be ≤ 3%
Calibration Blank	Immediately after each initial calibration (ICB), every 10 samples (CCB), and at the end of the analytical sequence	≤ PRL	If criteria are not met, follow the corrective actions in 10.3.2. If the concentration in the blank is outside the control limit and <1/10 the concentration in the sample, the sample result may be reported.
CRDL Standard	Once per ICAL, immediately after verification	Each element tested must be at least between 50-150% of the PRL value. If reporting for the state of MN, limits of 60-140% are applied.	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.3.4 *Pace Metals 3P Team determined it is a best practice to use PRL limits of at least 50-150%. Limits may be more strict if required by client.
ICSA	Once per ICAL, immediately after verification	Results for non-interference elements = ND ± 2x RL Recovery of interfering elements must be 80-120% of true value	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.3.5
ICSAB	Once per ICAL, immediately after ICSA if requested by the client.	Results for non-interference elements = True Value ± 20%.	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.3.5
Continuing Calibration Verification	After every 10 samples and at the end of analytical sequence	≤±10% of true value The % RSD between replicate integrations of a CCV std must be <5% (min of 2 integrations).	If criteria not met, follow corrective actions in 10.3.6

10.4 Calibration Corrective Actions

10.4.1 Calibration Linearity Problems

10.4.1.1 Reanalyze the original calibration standards to determine instrument consistency.
10.4.1.2 Prepare and analyze a new calibration standard to determine preparation consistency / standard integrity.
10.4.1.3 Perform instrument maintenance, document in instrument log book
10.4.1.4 Recalibrate instrument according to procedures in Section 9.

10.4.2 Calibration Blank Problems

10.4.2.1 Reanalyze the original CB standard to determine instrument consistency.
10.4.2.2 Prepare and analyze a new CB standard to determine preparation consistency / standard integrity. Note: If you run two CB, only one may be used for reporting purposes.
10.4.2.3 Perform instrument maintenance, document in instrument log book
10.4.2.4 Recalibrate instrument according to procedures in Section 10.
10.4.2.5 Reanalyze any samples not bracketed by passing CBs.
10.4.2.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing CB that exceeded criteria may be reported if 10X concentration in the blank is less than the reported result.
Samples analyzed with a bracketing CB that exceeded criteria may be reported if the sample result is a non-detect.

10.4.3 Initial Calibration Verification (Secondary Source) Problems

10.4.3.1 Reanalyze the original ICV standard to determine instrument consistency.
10.4.3.2 Prepare and analyze a new ICV standard to determine preparation consistency / standard integrity.
10.4.3.3 Perform instrument maintenance, document in instrument log book
10.4.3.4 Recalibrate instrument according to procedures in Section 10.
10.4.3.5 Reanalyze any samples not bracketed by passing ICVs.
10.4.3.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

10.4.4 Exceptions: Samples analyzed with a bracketing ICV that exceeded criteria due to an increase in response may be reported if the target analytes were not detected in the samples.

10.4.5 CRDL Standard Problems

10.4.5.11 Check CAL0 – Elevated counts resulting from contamination may cause the CRDL to fail low
10.4. 5.2 Reanalyze the original CRDL standard to determine instrument consistency.
10.4.5.3 Prepare and analyze a new CRDL standard to determine preparation consistency / standard integrity.
10.4.5.4 Perform instrument maintenance, document in instrument log book
10.4.5.5 Recalibrate instrument according to procedures in Section 10.

10.4.4.5 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with CRDL that is outside criteria may be footnoted or the CRDL for the reported sample adjusted.

10.4.6 ICSA/ICSAB Standard Problems

10.4.5.1 Reanalyze the original **ICSA/ICSAB** standard to determine instrument consistency.
10.4.5.2 Prepare and analyze a new **ICSA/ICSAB** standard to determine preparation consistency / standard integrity.
10.4.5.3 Adjust IEC and rerun **ICSA/ICSAB.** To update IECs, follow the instruction below.
10.4.5.4 The Interelement Correction Factors (IEC's) are used to correct for false signals (positive and negative) produced when peaks overlap in the spectra.
10.4.5.5 To set up Initial IEC, run single element standards with only background correction points in place. The standards are run at the linear range.

The following calculation is done:

$$\frac{\text{Element result}(\mu g/L)}{\text{Interfering element result}(\mu g/L)} = \text{IEC}$$

10.4.5.6 This ratio is entered in the IEC table in the method file. Type the ratio under the k1 header in the appropriate row. Enter a negative before the ratio if it is a negative interference.
10.4.5.6.1 Perform instrument maintenance, document in instrument log book
10.4.5.6.2 Recalibrate instrument according to procedures in Section 9.

10.4.7 Continuing Verification Problems

10.4.6.1 Reanalyze the original CCV standard to determine instrument consistency.
10.4.6.2 Prepare and analyze a new CCV standard to determine preparation consistency / standard integrity.
10.4.6.3 Perform instrument maintenance, document in instrument log book
10.4.6.4 Recalibrate instrument according to procedures in Section 10.
10.4.6.5 Reanalyze any samples not bracketed by passing CCVs.
10.4.6.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing CCV that exceeded criteria due to an increase in response may be reported if the target analytes were not detected in the samples.

11. PROCEDURES

11.1 Aqueous Sample Preparation for Total or Dissolved Metals by 3010A – Hot Block Digestion

11.1.1 Groundwater samples for dissolved metals may be analyzed without digestion

unless there is visible sediment. Samples contains visible sediment must be digested. All other water samples must be digested.

11.1.2 Aqueous samples for total or dissolved metals (if visual precipitation) may be prepared according to EPA 3010A. Samples for dissolved metals must be filtered prior to preservation. Once preserved, samples should be allowed to sit for 24 hours prior to preparation. TCLP/SPLP/ASTM extracts do not require to sit for 24 hours prior to digestion.

11.1.3 Verify that the hood is turned on. Turn on the Hot Block and set the temperature so that samples on the block maintain a temperature of 95 +/- 5ºC. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the sample temperature in the sample preparation logbook. The well position should be rotated on a per batch basis to ensure that the block temperature is verified across the block.

11.1.4 Place a 50mL well-mixed aliquot of each sample in digestion vials. Place 50 mL of Nano-pure water into digestion vials for each MB and LCS (one per 20 samples). Prepare the MS and MSD by placing 50mL aliquots of selected sample from the batch into digestion vials. One MS and one duplicate or MSD must be prepared for each analytical batch of 20 samples or less. Additionally, one MS must be prepared for each TCLP/SPLP/ASTM sample matrix digested in a digestion batch.

11.1.5 Spike the LCS, MS, and MSD with 1.0mL of the spiking solution.

11.1.6 Carefully add the digestion reagents to each sample, MB, LCS, MS, and MSD vial.

Digestion Program for all Aqueous Samples, EPA Method 3010A

Steps	Reagents[1]	Actions
1	5.0mL concentrated HNO_3, 2.5 mL HCl	Heat at 95 +/- 5ºC °C for 4 hours
2		Remove digestates from Hot Block and allow to cool in the hood.
3		Adjust the final volume of each vial to 50 mL by diluting with 18 MOhm Nano-Pure Nano-pure water. Place on screw cap and shake thoroughly.
4		Centrifuge, let settle, or filter sample only if there is concern that insoluble materials may clog the nebulizer. Filtering is liable to cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned and pre-rinsed with dilute HNO_3. If filtration is required, filter all QC samples as well.

1. Acid ratios must be maintained to match method. Those specified are based on a 50mL sample volume

Note: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the samples and re-prep.

11.1.7 Record the lot numbers of all reagents and materials in the digestion logbook. This includes the lot numbers of the digestion tubes, filters or filter paper and any additional materials in contact with the sample matrices.

11.2 Soil/ Solid/Sludge/Paint Chip/Wipe Sample Preparation by 3050B

11.2.1 Soil, sludges, and solids are prepared according to Method 3050B. Wipes and Paint Chips are also prepared using 3050B.

11.2.2 Verify that the hood is turned on. Turn on the Hot Block and set the temperature to 95±5ºC. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the sample well ID and temperature in the sample logbook. This well measurement should be rotated on a per batch basis to ensure that the block temperature is consistent across the block.

11.2.3 To prepare Paint Chips for digestion, transfer paint chips into labeled drying tins. Dry samples at 103-105 degrees Celsius for 12-24 hours. Remove samples from oven and desiccate for 1 hour. Paint chips requirement that an LSC/LCSD be prepared per batch.

11.2.4 If possible, mix each sample thoroughly to homogenize. If homogenization is not feasible, remove the top layer of the sample from the container and discard. Remove a sample aliquot in the range of 0.5 – 0.599 g for analysis (0.5g dry sample). Weigh the aliquot to the nearest 0.01g and transfer to a digestion vial. Refer to Pace S-ALL-Q-021, *Sub-Sampling* SOP, for more details. For paint chips, Weigh out 0.5 gram of sample into a digestion tube. If the sample available is less than 0.5 gram, use the entire sample. For Wipes, digest the WHOLE wipe in the digestion vial. For sludges with % solids >10% and <50%, weigh out 2.5-5 grams.

11.2.5 Prepare the MB and LCS by adding 2.0 mL of Nano-pure water into digestion vials containing 0.500-0.504g of Teflon chips. The weight is documented as 0.500g so as to not bias the results. Prepare the duplicate, MS and MSD by placing aliquots of a selected sample from the batch into digestion vials as described for environmental samples. Spike the LCS, MS, and MSD with 1.0 mL spiking solution.

11.2.6 Carefully add the digestion reagents to each sample, MB, LCS, MS, and MSD vial and heat them with the Hot Block at 95±5ºC according to the following digestion program.

11.2.7 MS/MSDs are not performed on paint chips, because the sample concentration is frequently excessively high and dwarfs any spike. As a result an LCSD is performed for paint chip batches...

Digestion Program for Soil/ Solid samples by method 3050B

Steps	Reagents[1]	Actions
1	Nano-pure water	Add Appoximately 2.0 mLs of water to all vessels in the prep batch.
2	10.0 mL 1:1 HNO3	Cover with watch glass and heat without boiling for 2 hours. Remove from heat and cool.
3	2.5 mL 30% H_2O_2	Cover with watch glass and heat on Hot Block for 15 minutes or until effervescence subsides. Warming will start the peroxide reaction. Care must be taken to ensure that losses do not occur due to vigorous effervescence. Remove and allow to cool.
4	2.5mL concentrated HCl	Reflux on Hot Block until reaction is complete (no brown fumes) without boiling. Allow samples to cool.
5		Dilute sample to 50-mL final volume with 18 MOhm Nano-Pure water. Place screw cap on vials, shake, allow sample to settle, centrifuge at 2000-25000 rpm for 10 minutes, OR and filter if necessary.

1. Acid ratios must be maintained to match method. Those specified are based on a 0.5g sample amount
2. In effect, this produces the 1:1 nitric acid (required by method 3050) in the actual digestion cup.

11.2.8 Record the lot numbers of all reagents and materials in the digestion logbook. This includes the lot numbers of the digestion tubes, filters or filter paper and any additional materials in contact with the sample matrices.

11.3 ICP System Preparation

11.3.1 Preventative Maintenance

Inspect the sample introduction system including the nebulizer, torch, injector tube and up-take tubing for salt deposits, dirt and debris that could restrict solution flow and affect instrument performance. Document all maintenance in ICP maintenance logbook.

See attachment I for troubleshooting guide.

11.3.2 Operating Parameters

Set up and configure the ICP according to manufacturer's operating instructions using operating parameters shown in the following table.

Table 11.3.1 - Operating Parameter for ICP Systems

Operating Parameter	ICAP 6500 20073913
Argon Flow	80 L/min
Nebulizer Pressure	0.55 L/min
RF Output	1175 W
Pump Speed	15 rpm
Aux Gas Flow	0.5 L/min
Coolant Gas Flow	15 l/min

11.3.3 Calibrate ICP according to Section 10.

11.4 Sample Analysis

11.4.1 Batch Sequence

If using an autosampler, enter an autosampler sequence into the ICP data system per manufacturer's instructions. Calibration blanks, standards, initial checks, and interlement correction standards shall be run before analysis of environmental samples. All samples, including calibration, checks, and other standards, can also be run manually.

Table 11.4.1 – Example analytical sequence after calibration of a blank and a standard.

Autosampler Position	Sample Description	Autosampler Position	Sample Description
1	ICV	27	Sample 15
2	ICB	28	Sample 16
3	CRDL	29	CCV
4	ICSA*	30	CCB
5	CCV	31	Sample 17
6	CCB	32	Sample 18
7	MB1	33	Sample 19
8	LCS1	34	Sample 20
9	Sample 1	35	MB2
10	Sample 2	36	LCS2
11	Sample 3	37	Sample 21
12	Sample 3 Matrix Spike	38	Sample 22
13	Sample 3 Matrix Spike Duplicate	39	Sample 23
14	Sample 4	40	Sample 24
15	Sample 5	41	CCV
16	Sample 6	42	CCB
17	CCV	43	Sample 25
18	CCB	44	Sample 26
19	Sample 7	45	Sample 27
20	Sample 8	46	Sample 28
21	Sample 9	47	Sample 28 Matrix Spike
22	Sample 10	48	Sample 28 Matrix Spike Duplicate
23	Sample 11	49	Sample 29
24	Sample 12	50	Sample 30
25	Sample 13	51	CCV
26	Sample 14	52	CCB

* ICSAB will follow the ICSA if needed/requested by client.

11.4.2 Load Autosampler

11.6.1.1 Load the autosampler tray according to the vial position on the sequence table.

11.5 **Sample Analysis**

11.5.1 Analyze all standards, quality control samples, and environmental samples.

11.6 **Data Reduction**

11.6.1 **Quantitative Analysis –** The instrument produces results in µg/L. If the initial sample aliquot and final digestate volumes are the same, the ICP data system will calculate the concentration of each element directly. If the initial and final volumes are different, the actual values are posted into the LIMS system and calculations are performed in the LIMS.

11.6.1.1 Water Samples – Since initial sample aliquot and final digestate volumes are the same, the iCAP analyzer data system will calculate the concentration directly. No further calculations are necessary unless the sample was diluted.

11.6.1.2 Soil/Solid/Paint Chip/Sludge Samples -

Final Result (mg/kg dry weight corrected) =

$$\frac{\text{Raw data result (µg/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{Sample weight (g)} * \text{Dry weight for soil/solid (decimal form)}}$$

11.6.1.3 Wipes

Final Result (µg/wipe) =

$$\frac{\text{Raw data result (µg/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{1 wipe}}$$

11.6.2 Samples with analyte concentrations that exceed 90% of the upper linear range must be diluted and re-analyzed.

11.6.3 Any sample in which any element value is below the instrument detection limit or PRL must be reported as less than the PRL (or ND) for that specific element. Do not report data below the element PRL concentration unless it is qualified as an "estimated" result with a "J" or "B" data qualifier.

11.6.4 Metals Analytical Review and Reporting System (ADAMS) or LimsLink is used to transfer data from the instrument to the LIMS system.

12. QUALITY CONTROL

12.1 Refer to the most current version of the Pace Quality Manual Appendix I Quality Control Calculations and SOP: S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

12.2 Instrument Quality Control

12.3 Refer to Table 11.2 for initial and continuing calibration criteria and corrective actions. This includes ICV, CCV, CB, PRL, and ICSA standard checks.

12.4 Batch Quality Control

Table 12.2 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Nano-pure water	One per 20 samples	1) Target analytes must be less than the RL.	1) Re-analyze blank to confirm failure. 2) Qualify results and / or re-extract associated samples. **Exceptions:** 1) If sample ND, report sample without qualification 2) If sample result >10x MB detects no qualification is needed... 3) If sample result <10x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Nano-pure water and Spiking solution (see table 10.3.3 for elements)	One per 20 samples	Laboratory Calculated Limits or default limits of 80-120% Should not exceed 80-120%	1) Reanalyze the LCS to verify failure 2) If LCS passes, review samples for potential problems 3) If problem persists, check spike solution 4) Re-digest and re-analyze samples where possible **Exceptions:** 1) If LCS rec > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Environmental Sample and Spiking solution (see table 10.3.3 for elements)	One per 20 samples. For leached samples one per sample matrix (soil and wood chips would each require a separate matrix spike).	Laboratory Calculated Limits or default limits of 75-125% Should not exceed 75-125%	1) If LCS and MBs are acceptable, the MS may be reported with appropriate footnote indicating matrix interferences
MSD / Duplicate	MS Duplicate OR (alternative) Sample Dup	A duplicate sample OR MS/MSD pair must be analyzed per batch of 20 samples.	Laboratory Derived Limits for RPD or default limits of 20%. Recovery criteria should not exceed 75-125%.	1) Results not meeting criteria must be footnoted on client report.

12.5 Dilution Test – A dilution test is recommended but not required for method 6010B. A dilution test is run only at client request.

12.5.1 Perform a 5X dilution on one sample per preparation batch. Diluted and undiluted sample results should agree within 10% of each other if the sample contains analyte concentrations at least 50 times the IDL. If agreement between diluted and undiluted samples meeting the concentration requirements is >10%, then matrix effect should be suspected and samples footnoted appropriately.

12.6 Post-digestion Spike Test or Analyte Addition – Post digestion spikes are recommended but not required for method 6010B. A post spike is run only at client request.

12.6.1 Spike one digestate from a preparation batch to achieve the same analyte addition level as found in the LCS. The acceptable recovery range is 75-125%. If outside this range, matrix effects should be suspected and samples annotated.

12.7 **Internal Standards –** Yttrium is used as an internal standard. Yttrium is not calibrated for. Precision of the Yttrium reps are evaluated using < or = 5% RSD. Accuracy of the Yttrium average count levels are evaluated using 50-150% recovery as compared to the calibration blank. If either of these criteria is not meet, the sample or quality control standard must be rerun, possibly at a dilution.

12.8 **Multiple Instrument Intregration RSD** – The RSD is required to be evaluated between multiple instrument integrations. The RSD must be less than or equal to 20% if the analyte concentration is greater than the reporting limit. If the RSD is greater than 20%, the laboratory must reanalyze the sample.

13. METHOD PERFORMANCE

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted annually per S-GB-Q-020, *Determination of LOD and LOQ* per instrument.

13.2 Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures*.

13.3 Linear Dynamic Range Study

13.3.1 It is a Pace best practice to perform this linear dynamic range study determination once, keep the data, and then annually or semi-annually verify with a single high point. If additional range is to be considered, the laboratory must perform the study as indicated below.

13.3.2 A linear dynamic range study must be conducted at least annually. The study is conducted for each element by analyzing increasing concentrations (at least 3 levels) until the results generated exceed ±10% difference from the true value. The highest concentration within the 10% criteria is the maximum of the linear range for that element.

13.4 **Instrument Detection Limits (IDLs)** in µg/L are estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined at least quarterly.

13.5 **Method Modifications**

Method modifications for EPA method 6010B are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- Procedures identified as “Best Practices” by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- When insufficient volume for the method specified matrix spike/matrix spike duplicate (MS/MSD) is not provided by the client, a laboratory control spike duplicate will be analyzed to demonstrate precision criteria. Laboratory batches will be qualified with the appropriate “M5” data qualifier. When performing this analysis on paint chip samples, a MS/MSD will not be completed on the samples due to high levels of elements present in the native sample.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management*.

14.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15. REFERENCES

15.1 USEPA, SW-846, Method 6010B, "Inductively Coupled Plasma Atomic Emission Spectrometry", December 1996.

15.2 USEPA, SW-846, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectrometry", December 1996.

15.3 USEPA, SW-846, Method 3050B, "Acid Digestion of Sediments, Sludges, and Soils", December 1996.

16. TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS, APPENDICES, ETC.

16.1 **Attachment I** - ICP Troubleshooting Guide

16.2 **Attachment II** – ICP Flowchart

17. REVISIONS

Document Number	Reason for Change	Date
S-ALL-GB-M-002-REV.00	Restructured document format to template. Added tables and other content to create an "SOP Template" in which local edits are allowed. Modified signature page to include department manager and SOP template number Updated SOP template number. Made editable material red Updated calibration section to reflect change for PRL requirements. Updated linear range to include best practice established by PaceMetals 3P team. Added separate sections for 3005 and 3010A digestion procedures. Updated procedures to match what is being performed in Wisconsin (noted in red) Added reference to Interelement Correction Factors and Instrument Detection Limits. Add a flowchart.	28Feb2007
S-ALL-GB-M-002-REV.01	Updated Section 2 to include iCAP Updated Table 3.1 to include iCAP 6500 wavelengths and current PRLs. Removed reference to Conifer2K in sections 3.4. In section 8, changed hours from preservation to preparation/analysis from 16 to 24. In section 9, changed instrument to iCAP 6500 from iCAP 61E Removed "(5% nitric and 5% hydrochloric acid)" from 10.3 Added Sr to tables 10.3.1, 10.3.2, 10.3.3, 10.3.4, 10.3.5, and 10.3.6b. Renamed Tables 10.3.5 to 10.3.7 to 10.36b to 10.3.7 to eliminate table number duplication. Removed instructions for As profile. Updated table 10.3.4 to use 1.0mL spike instead of 0.5 mL. Removed As Profile from 11.1. Added Carbon profile. Removed Se and Pb exception from IEC determination in 11.4.3.24	19Nov2007
S-ALL-GB-M-002-REV.02	Updated signature Page Deleted Section 7. Renumbered document. Section 9.2 – Updated Inter-element correction solution (ICSAB) description for the Green Bay Laboratory. Section 11.1.1 – Digestion criteria updated. Section 11.2.3 – Paint chips LCS/LCSD requirement added. Table 12.2 – Changed the Method Blank acceptance criteria from 20x to 10x. Section 13 – Updated SOP references.	08Dec2008
S-GB-M-014-Rev.00	Updated Signature Page Renumbered document following SOP: S-ALL-Q-003 *Document Numbering* Table 3.1: Updated Pb RL to 7.5 Section 9.2.1 and 10.2: Added that calibration standards can be used until manufacturer's expiration date Section 11: Changed amount of HNO3 to 5mL from 3mL during digestion.	28Apr2010
S-GB-M-014-Rev.01	Added TCLP Criteria to body of document Corrected Formatting throughout document. Corrected Header information to 6010C from 6010B. Table 3.1: Updated Reporting Limits to match current. Table 3.1, Section 11.6.1.3: Changed unit for Wipe to Total µg. Section 7.1: Added TCLP/SPLP/ASTM Preservation Requirement and Holding Times. Section 7.2: Preservation Check: Changed maximum volume of acid to add as 2% from 5%. Table 9.3.6: Updated Vendor and Catalog numbers. Section 11.6.4: Updated reference to ADAMS From MARRs.	28Jun2011

Document Number	Reason for Change	Date
S-GB-M-014-Rev.02	Table 3.1: Updated TCLP/SPLP/ASTM PRLs. Section 9 Tables: Updated formatting to merged cells when the same standard, acids, final volume, etc. are used for one complete standard. Section 9: Updated expiration dates of standards to 6 month or manufacturer expiration date, whichever is sooner. Added Table 9.3.5 CRDL Intermediate Standard. Section 13.4: Changed requirement for IDL Studies to quarterly from yearly. Section 13.6.4 and 13.6.5: Added method modifications. Throughout document: Updated instances of Reagent Water to Nano-Pure Water, updated RLVS to CRDL standard	30Sep2011
S-GB-M-014-Rev.03	Section 4.2: Updated language on physical interferences. Section 4.5: Updated high salt interferences language. Table 9.3.2: Updated volume of standards used. Table .9.3.5: Updated K concentration in stock and working std. Table 9.3.6: Updated Ag concentration in stock and working std. Section 11.2.4: Updated digestion procedures	07Dec2012

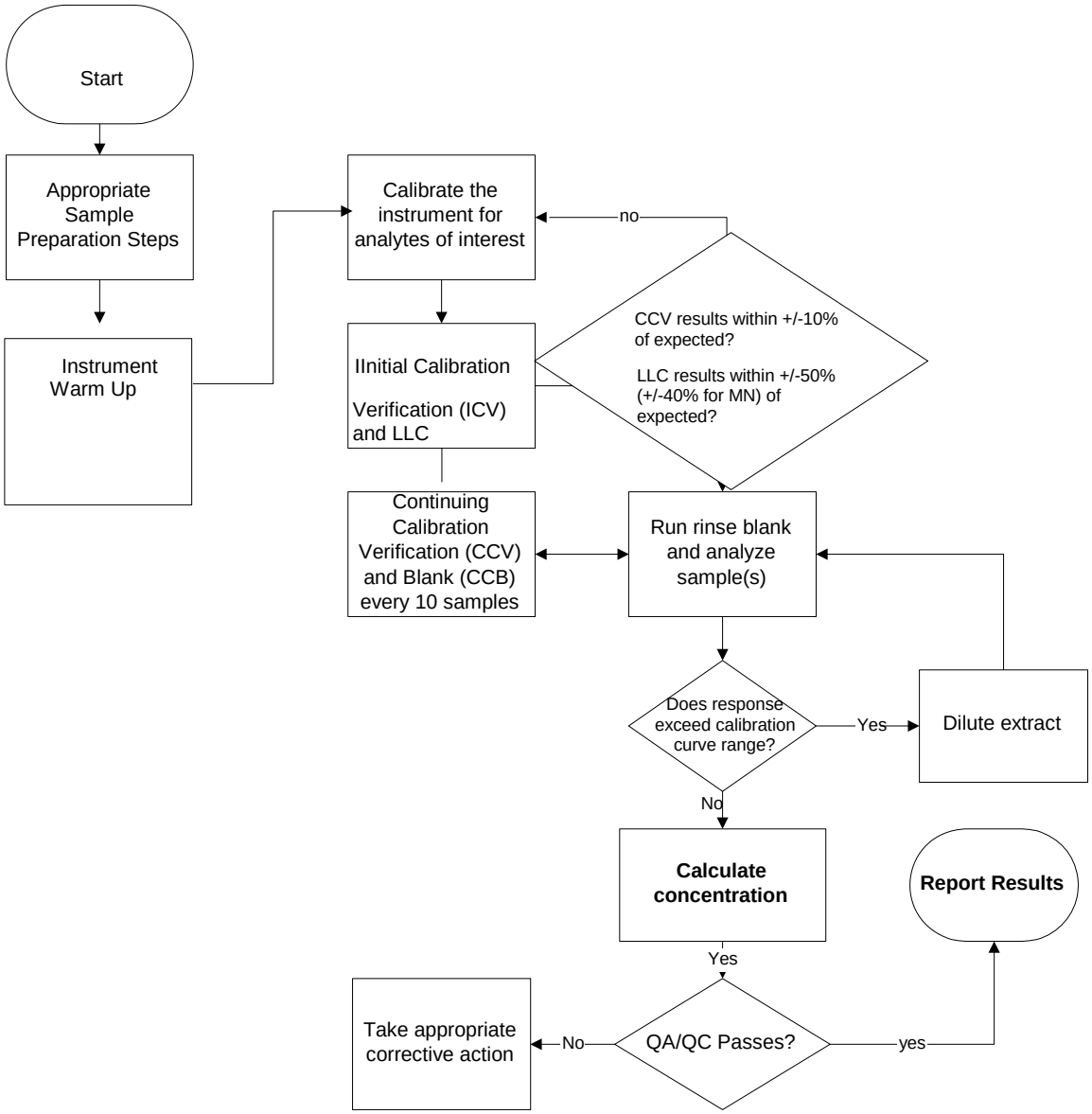
Attachment I

GENERAL ICP TROUBLESHOOTING GUIDE

Problem	Solution
Poor linearity in a dilution series of standards.	This may be as easy as not having spiked your standards correctly. Before you remake your standards, first examine the sample introduction area, making sure you're getting a consistent flow of sample through your pump tubing and that your connectors are not too tight. Then check your nebulizer and injector for visible build up. If build up is present, clean in a dilute nitric acid (HNO_3) solution for about 10-15 minutes. Also examine your torch, making sure it is also reasonably clean. Lastly, check the argon tank to determine that it is not running on empty. If everything is satisfactory, remake standards. If problems persist, try a second source of standards to determine if your initial set of standards has gone bad.
Bubbles are collecting in the back area of the nebulizer (glass type).	The reason why bubbles form in this back area is because the pump tubing is not inserted deeply enough into the cavity of the nebulizer. Cut the tip of the pump tubing to be inserted into the nebulizer at a roughly 45º angle, preferably with a razor blade to obtain a nice, clean cut. Dip the cut end into a wetting agent such as dilute Triton X solution, and start to insert into the nebulizer. Using moderate pressure, begin to work the pump tubing into the nebulizer while twisting the tubing. The twisting action causes the tubing to gradually decrease in diameter, making it easier to insert deeply into the cavity.
Hard instrument failure (software related). These are generally problems associated with the software communication with the instrument.	Exit out of the program. Shut down the PC that is linked to the instrument itself and restart. During the PC reboot process, locate the reset button(s) on the front and/or back of the instrument and depress them. Enter back into the program and continue your analysis. This generally will fix this type of problem, but if you encounter additional problems of this type, then a service call may be necessary for more advanced troubleshooting advice.
Peristaltic pump occasionally stutters or stops. This problem may be hard to notice since this is a gradual wearing of the belt that attaches to the pump motor and turns the rollers themselves.	Replace the belt if you have an available spare. If you do not have a replacement, a service call needs to be made. A service person will generally come out and install the belt but on occasion, based on your mechanical abilities, they may ship the part to you and have you perform the installation.

Attachment II

ICP Flowchart



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STANDARD OPERATING PROCEDURE

The Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy – CETAC M-7500

Reference Methods: EPA SW-846 Methods 7470A and 7471A/B

SOP NUMBER:	S-GB-M-017-REV.01
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APPROVAL

Nils Melberg, Laboratory General Manager — Date: 04/09/13

Kate Grams, Laboratory Quality Manager — Date: 4/8/13

Chad Rusch, Department Manager — Date: 04/08/2013

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. PURPOSE/IDENTIFICATION OF METHOD

1.1 The purpose of this Standard Operating Procedure (SOP) is to describe the determination of mercury in water, soil/solid samples, and biological tissue analyzed by the Cetac M-7500 instrument.

2. SUMMARY OF METHODS

2.1 Cold vapor atomic absorption utilizes the volatile property of elemental mercury at the 253.7 nm wavelength. To release mercury from organic complexes, the sample is digested with oxidizing reagents and acids in a hot block digester. After digestion, the oxidizing reagents are neutralized. Stannous chloride is added to reduce ionic mercury to the ground state. The Flow Injection Analysis System sweeps the volatile elemental mercury out of the sample and into the cell of an atomic absorption spectrophotometer. The absorbance signal is proportional to the amount of mercury in the sample.

3. SCOPE AND APPLICATION

3.1 This method is applicable to water samples (including drinking water, surface water, and domestic and industrial wastewaters), solid samples (including soils, sediments, sludges, and, solid wastes), wipes, TCLP, SPLP and ASTM leachates, and biological tissue samples.

3.2 This procedure is restricted to use by, or under the supervision of, analysts experienced in the digestion of samples for metals analysis and analysis of digestates by atomic absorption spectrometry. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. APPLICABLE MATRICES

4.1 This SOP applies to aqueous, solid, and biological tissue samples.

5. LIMITS OF DETECTION AND QUANTITATION

5.1 All current MDLs and RLs are listed in the LIMS and are available by request from the Quality Manager.

6. INTERFERENCES

6.1 Samples can contain diverse matrix types, each of which may present analytical challenges. Spiked samples and Laboratory Control Samples are important for determining digestion efficiency.

6.2 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide, as NaS, do not interfere with recovery of added inorganic Mercury from D.I. water.

6.3 Copper has also been reported to interfere; however, copper concentrations as high as 10mg/L had no effect on recovery of Mercury from spiked samples.

6.4 Samples high in chlorides require up to 7.5 mL of additional potassium permanganate. During the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the Mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent. Samples may be diluted to decrease the chlorides. Alternatively, the sample may be allowed to stand for at least an hour under a hood (without active purging) to remove the chlorine.

6.5 Certain volatile organic materials that absorb at this wavelength may also interfere. A preliminary run without reagents would determine if this type of interference is present.

7. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1 BIOTA SAMPLES

7.1.1 Biota samples can be collected in clean plastic or glass containers or plastic baggies.

7.1.2 Biota samples are kept frozen until time of preparation to preserve integrity. Hold time is 28 days from removal from freezer.

7.2 SOIL SAMPLES

7.2.1 Soil and waste samples should be collected in clean wide-mouth glass containers to facilitate obtaining representative aliquots for measurement.

7.2.2 Samples should be stored at 4±2°C and analyzed as soon as possible to minimize microbiological decomposition of organic solids. Samples must be analyzed within 28 days of collection.

7.3 WIPE SAMPLES

7.3.1 Wipe should be collected in clean wide-mouth glass containers.

7.3.2 Samples should be stored at 4±2°C and analyzed as soon as possible to minimize microbiological decomposition of organic solids. Samples must be analyzed within 28 days of collection.

7.4 WATER SAMPLES

7.4.1 Water samples may be collected in plastic or glass containers and must be preserved with HNO_3 to pH <2 and stored at room temperature, acid not to exceed 2% of the estimated sample volume.

NOTE: Aqueous samples that react violently to the addition of acid may be collected without chemical preservation with proper variances approved by the regulatory authority. The responsibility of requesting this variance lies with the sample collector.

NOTE: Samples may be preserved in the lab. The samples may not be processed until 24 hours after preservation with a pH test of <2.

7.4.2 Samples must be analyzed within 28 days of collection.

7.5 TCLP, SPLP, and ASTM SAMPLES

7.5.1 Leach extracts should be **collected in plastic or glas**s containers and must be refrigerated at 4±2 ^{o}C until time of preservation with HNO_3 to pH <2 and then stored at room temperature, acid not to exceed 2% of the estimated sample volume.

NOTE: TCLP Samples are not required to sit for 24 hours prior to digestion after the addition of nitric acid.

7.5.2 Samples must be extracted within 28 days from field collection to leach extraction. Samples must be digested and analyzed within 28 days of the leach extraction. Total elapsed hold time is 56 days.

8. DEFINITIONS

8.1 Definitions of terms found in this SOP can also be found in the Pace Quality Manual. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP or the Pace Analytical Services' Quality Manual Glossary, Section 10.0.

8.2 Biota Control Blank (Matrix Blank) - A sample of a matrix that is used for the control spike. The biota control blank will either be catfish, tilapia, chicken, or other tissue whichever is available at the time of analysis. The biota control blank should be "farm-raised" to minimize background mercury levels. The concentration in the biota control blank will be subtracted from the concentration of the laboratory control spike. This is done because the biota control blank is known to have some contamination. This SOP will reference biota control blank for ease. Note: For plant material analysis, alfalfa can be used as the biota control blank matrix.

8.3 Reagent Grade – Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents, which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

8.4 Standardized Reference Material (SRM) – A certified reference material produce by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. A SRM is analyzed with each analytical batch of biota samples.

8.5 Pace Reporting Limit (PRL) – The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e. statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.

9. EQUIPMENT AND SUPPLIES (INCLUDING COMPUTER HARDWARE AND SOFTWARE)

9.1 Hot Block – Operated at 95 $\pm$ 5°C. Heating block should be Environmental Express "Hot Block", or equivalent.

9.2 Digestion Vials – Clean 15- mL screw cap vials, Environmental Express (SC415), or equivalent.

9.3 Digestion Vials- Clean 68-mL screw cap vials, Environmental Express (SC475), or equivalent.

9.4 Analytical Balance – Capable of weighing to 0.01g.

9.5 Volumetric Flasks – Assorted, Class A

9.6 Graduated Cylinder – 50-mL

9.7 Mechanical Pipettes – Assorted adjustable air-displacement pipettes with disposable tips (Eppendorf or equivalent).

9.8 pH Strips

9.9 CETAC M-7500 with auto-sampler

9.10 Inert Sparging and Carrier Gas – Argon

9.11 Autosampler vials

9.12 Autosampler Pump Tubing

9.13 Specimen Cups

9.14 DORM-4 or equivalent

9.15 Analytical Balance – Capable of weighing to 0.0001g.

10. REAGENTS AND STANDARDS

10.1 Trace Metals Water

10.2 Nitric Acid (HNO_3) – 9598-34 J.T. Baker, or equivalent.

10.3 Hydrochloric Acid (HCl) – 9530 –33 J.T. Baker, or equivalent.

10.4 Sulfuric Acid (H_2SO_4) – 9681-33 J.T. Baker, or equivalent.

10.5 Potassium Permanganate ($KMnO_4$) solution (5%) – Dissolve 50.0 g of $KMnO_4$ into ~800 mL of trace metals water. Dilute to 1000 mL. Prepare fresh each month.

10.6 Potassium Persulfate ($K_2S_2O_8$) solution (5%) – Dissolve 50.0 g of $K_2S_2O_8$ into ~800 mL of trace metals water. Dilute to 1000 mL. Warm to dissolve. Prepare fresh each month.

10.7 Hydroxylamine Hydrochloride ($NH_2OH \bullet HCl$) Solution (12%) – Dissolve 120 g NaCl and 120g $NH_2OH \bullet HCL$ in ~800 mL of trace metals water. Dilute to 1000 mL. Prepare fresh each month.
Stannous Chloride Solution ($SnCl_2$) - Dissolve 100.0 g of SnCl in ~ 800 mL of trace metals water. Add 70 mL of HCL. Dilute to 1000 mL. Discard if precipitate forms.

10.8 Acid Rinse Solution- Add 100 mL of HCl and 40 mL concentrated Nitric acid
In ~1000 mL of trace metals water dilute to 2000 mL.

10.9 Analytical Standards – Standard solutions are required calibration, calibration checks, and sample spiking solutions. The following describes the contents of each type of solution. The commercial sources for stock solutions, recipes for preparing dilutions and working standards, and concentrations in standard solutions are presented in Table 1, Table 2, and Table 3.

Table 1
Stock Standards

Standard	Concentration	Vendor	Catalog #
Hg Calibration Stock	1000µg/mL	High-Purity Standards	CGHG1-1
ICV Calibration Stock	1000µg/mL	Inorganic Ventures, Inc.	100033-1

Standards are used until they expire based on the manufacturer's expiration date.

Table 2
Intermediate and Calibration Standards
Waters

Standard	Stock Standard	Conc.	Amount Used	Amount of conc. HNO_3 used	Final Volume (with trace metals water)	Final Conc.
Hg ICV Stock	-	1000µg/mL	-	-	-	1000 µg/mL
Hg Calibration Intermediate	Hg Calibration Stock	1000µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg ICV Intermediate	Hg ICV Stock	1000 µg/ml	20 µL	6.0 mL	200 mL	100 ppb
Hg Cal. 1	-	-	-	-	10 mL	0.0 ppb
Hg Cal. 2 and PRLs	Hg Cal. Intermediate	100 ppb	20 µL	-	10 mL	0.2 ppb
Hg Cal. 3	Hg Cal. Intermediate	100 ppb	100 µL	-	10 mL	1.0 ppb
Hg Cal. 4	Hg Cal. Intermediate	100 ppb	250 µL	-	10 mL	2.5 ppb
Hg Cal. 5	Hg Cal. Intermediate	100 ppb	500 µL	-	10 mL	5.0 ppb
Hg Cal. 6	Hg Cal. Intermediate	100 ppb	1000 µL	-	10 mL	10.0 ppb
Hg ICV	Hg ICV Intermediate	100 ppb	400 µL	-	10 mL	4.0 ppb
Hg CCV	Hg Cal. Intermediate	100 ppb	500 µL	-	10 mL	5.0 ppb
Hg ICB/CCB	-	-	-	-	10 mL	0.0 ppb

All intermediate dilution solutions must be prepared as needed, or at least every 7 days. Working solutions must be prepared fresh daily. Solutions may be stored at room temperature.

Table 3
Intermediate and Calibration Standards
Soils/Biota

Standard	Stock Standard	Conc.	Amount Used	Amount of conc. HNO_3 used	Final Volume (with trace metals water)	Final Conc.
Hg ICV Stock	-	1000µg/mL	-	-	-	1000 µg/mL
Hg Calibration Intermediate	Hg Calibration Stock	1000µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg ICV Intermediate	Hg ICV Stock	1000 µg/ml	20 µL	6.0 mL	200 mL	100 ppb
Hg Cal. 1	-	-	-	-	10 mL	0.0 ppb
Hg Cal. 2 and PRLS	Hg Cal. Intermediate	100 ppb	20 µL	-	10 mL	0.2 ppb
Hg Cal. 3	Hg Cal. Intermediate	100 ppb	100 µL	-	10 mL	1.0 ppb
Hg Cal. 4	Hg Cal. Intermediate	100 ppb	250 µL	-	10 mL	2.5 ppb
Hg Cal. 5	Hg Cal. Intermediate	100 ppb	500 µL	-	10 mL	5.0 ppb
Hg Cal. 6	Hg Cal. Intermediate	100 ppb	1000 µL	-	10 mL	10.0 ppb
Hg ICV	Hg ICV Intermediate	100 ppb	400 µL	-	10 mL	4.0 ppb
Hg CCV	Hg Cal. Intermediate	100 ppb	500 µL	-	10 mL	5.0 ppb
Hg ICB/CCB	-	-	-	-	10 mL	0.0 ppb

All intermediate dilution solutions must be prepared as needed, or at least every 7 days. Working solutions must be prepared fresh daily. Solutions may be stored at room temperature.

Table 4
Intermediate and Calibration Standards
Biota

Standard	Stock Standard	Conc.	Amount Used	Amount of conc. HNO_3 used	Final Volume (with trace metals water)	Final Conc.
Hg ICV Stock	-	1000µg/mL	-	-	-	1000 µg/mL
Hg Calibration Intermediate	Hg Calibration Stock	1000µg/mL	20 µL	6.0 mL	200 mL	100 ppb
Hg ICV Intermediate	Hg ICV Stock	1000 µg/ml	20 µL	6.0 mL	200 mL	100 ppb
Hg Cal. 1	-	-	-	-	30 mL	0.0 ppb
Hg Cal. 2 and PRLS	Hg Cal. Intermediate	100 ppb	60 µL	-	30 mL	0.2 ppb
Hg Cal. 3	Hg Cal. Intermediate	100 ppb	300 µL	-	30 mL	1.0 ppb
Hg Cal. 4	Hg Cal. Intermediate	100 ppb	750 µL	-	30 mL	2.5 ppb
Hg Cal. 5	Hg Cal. Intermediate	100 ppb	1500 µL	-	30 mL	5.0 ppb
Hg Cal. 6	Hg Cal. Intermediate	100 ppb	3000 µL	-	30 mL	10.0 ppb
Hg ICV	Hg ICV Intermediate	100 ppb	1200 µL	-	30 mL	4.0 ppb
Hg CCV	Hg Cal. Intermediate	100 ppb	1500 µL	-	30 mL	5.0 ppb
Hg ICB/CCB	-	-	-	-	30 mL	0.0 ppb

All intermediate dilution solutions must be prepared as needed, or at least every 7 days. Working solutions must be prepared fresh daily. Solutions may be stored at room temperature.

11. CALIBRATION AND STANDARDIZATION

11.1 A digested calibration curve is made up every time there are samples prepared. The curve is associated to the sample batch it is prepped with. Calibration requires analysis of a calibration blank and at least five levels of calibration solutions. The lowest calibration concentration is the pace reporting limit.

11.2 For the analysis of Biota, all calibration, instrument QC, Batch QC and samples are analyzed at a times 5 dilution. Since the calibration is also diluted at this level, the samples and QC do not need to be corrected for it.

11.3 The resultant correlation coefficient must be greater than 0.995. If this does not occur, then the instrument must be recalibrated and or the calibration curve and the associated samples must be re-prepped.

11.4 The calibration curve must pass an Initial Calibration Verification (ICV) that is analyzed after the calibration standards and before any samples. The ICV concentration is near the midpoint of the calibration curve and is made from a source other than the one used to make the calibration standards.

11.5 Pace Reporting Limit Standard (PRL or CRDL Std) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. When analyzing by 7471B, CRDLs must, at a minimum, bracket all samples and have a recovery of 70-130% of the true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

11.6 Every ten samples or less must be bracketed by Continuing Calibration Verifications (CCV). The CCV concentration is near the midpoint of the calibration curve.

11.7 The ICV and CCV limits are +/- 10% of their expected values. If one fails, then the instrument must be re-calibrated and any samples after the failure or not bracketed by an acceptable CCV, must be reanalyzed.

11.8 An acceptable Initial Calibration Blank (ICB), must be analyzed after the ICV.

11.9 Continuing Calibration Blanks (CCB) are analyzed after the CCVs.

11.10 The control limit for the ICB and CCB is the absolute value less than the Pace Reporting Limit. If one fails, then the instrument must be re-calibrated and any samples, after the failure or not bracketed by an acceptable CCB, must be reanalyzed.

12. PROCEDURE

12.1 Sample Preparation

12.1.1 Water samples

12.1.1.1 Verify preservation in the samples by checking their pH with a test strip (completed upon receipt in Sample Receiving Department).

12.1.1.2 Prior to analysis, the samples and the calibration curve must be digested.

12.1.1.3 Turn on the water bath and set the temperature to maintain a sample temperature of 95±5°C. Before placing samples in the water bath, verify that the water temperature is correct and adjust as required. Record the temperature in the sample logbook. Verify that the hood is functioning.

12.1.1.4 Add 10 mL of trace metals water minus the volume of standard to be added and add the appropriate amounts (as shown in Table 2) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.

12.1.1.5 Add 10 mL of trace metals water to a digestion vial for the Method Blank.

12.1.1.6 Add 10 mL of trace metals water and 0.5 mL of Hg ICV Intermediate to two digestion vials for the Laboratory Control Spike and Laboratory Control Spike Duplicate. Only prep LCSD if requested by client or there is not enough sample to run an MS/MSD.

12.1.1.7 Add 10 mL of each well-mixed sample to digestion vials.

12.1.1.8 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random or assigned by the client for every batch of 20 samples. An additional 10 mL of this sample along with 0.5 mL of Hg ICV Intermediate is added to two digestion vials.

NOTE: TCLP, SPLP, and ASTM extracts must be spiked prior to nitric acid preservation. One MS must be prepared per sample matrix (soil and wood chips would each require a separate matrix spike).

12.1.1.9 To each digestion vial add 0.25 mL of concentrated nitric acid. Swirl the digestion vials gently.

12.1.1.10 To each digestion vial add 0.5 mL of concentrated sulfuric acid. Swirl the digestion vials gently.

12.1.1.11 Add 1.5 mL of 5% $KMnO_4$, swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, an additional 1.5 mL of $KMnO_4$ solution is added to all samples, QC, and standards.

Note: If the sample still does not maintain color, discard applicable samples and dilute the samples prior to digestion.

12.1.1.12 Add 0.8 mL of 5% Persulfate solution. Place lid or cap on tube so as to allow pressure to vent but minimizing evaporation. Swirl the digestion vials gently.

12.1.1.13 Digest samples for 2 hours at 95±5°C.

12.1.1.14 Remove samples and let cool to room temperature.

12.1.1.15 Add 0.5 mL of 12% hydroxylamine hydrochloride solution. Swirl the digestion vials gently.

12.1.1.16 Samples are now ready for analysis.

12.1.2 Soil/Solid/ Wipe Samples

12.1.2.1 Prior to analysis the samples and calibration curve must be digested.

12.1.2.2 Turn on the hot block and set the temperature to maintain a sample temperature of 95+5°C. Before placing samples in the hot block to digest, verify that the water temperature is correct and adjust as required. Record the temperature in the electronic prep log. Verify that the hood is functioning.

12.1.2.3 Add 10 mL of 3% HNO3 minus the volume of standard to be added and add the appropriate amounts (as shown in Table 3) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.

12.1.2.4 Prepare one Method Blank by adding glass beads to it.

12.1.2.5 Prepare the Laboratory Control Spike (LCS) for soils, wipes, and wastes by adding glass beads to it. Then, add 0.5 mL of Hg ICV Intermediate to the digestion vial. Only prep a Laboratory Control Spike Duplicate (LCSD) if requested by client or there is not enough sample to run an MS/MSD. Prepare the LCSD in the same fashion as the LCS.

12.1.2.6 Weigh 0.3 g of homogenized sample into a labeled digestion vial. Record the weight to the nearest 0.01 g in the digestion prep log. For samples with high liquid content, a larger sample size may be used, as long as the digestion is complete. For wipes, put the WHOLE wipe in the digestion vial.

12.1.2.7 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random. Weigh 2 additional 0.3 g aliquots into digestion vials. Add 0.5 mL of Hg ICV Intermediate to each vial.

12.1.2.8 Add 10 mL of 3% HNO3 solution to all QC and samples. Swirl digestion vials to gently mix.

12.1.2.9 To each digestion vial add 0.5 mL of concentrated nitric acid. Swirl digestion vials to gently mix.

12.1.2.10 To each digestion vial add 1.0 mL of concentrated hydrochloric acid. Swirl digestion vials to gently mix.

12.1.2.11 Add 1.5 mL of 5% KMnO4, place lid or cap on tube so as to allow pressure to vent but minimizing evaporation, swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, an additional 1.5 mL of KMnO4 solution is added to all samples, QC, and standards. **Note: If the sample still does not maintain color, discard set and dilute the sample prior to digestion.**

12.1.2.12 Digest samples for 30 minutes at 95+5°C.

12.1.2.13 Remove samples and let cool to room temperature.

12.1.2.14 Add 1.5 mL of 12% hydroxylamine hydrochloride solution. Swirl digestion vials to gently mix.

12.1.2.15 Samples are now ready for analysis.

12.1.3 Biota Samples

12.1.3.1 Prior to analysis the samples and calibration curve must be digested.

12.1.3.2 Turn on the hot block and set the temperature to maintain a sample temperature of 95+5°C. Before placing samples in the hot block to digest, verify that the water temperature is correct and adjust as required. Record the temperature in the electronic prep log. Verify that the hood is functioning.

12.1.3.3 Add 30 mL of 3% HNO3 minus the volume of standard to be added and add the appropriate amounts (as shown in Table 4) of Hg Calibration Intermediate and Hg ICV Intermediate to digestion vials for the curve, ICV, CCV, ICB, and CCB.

12.1.3.4 Prepare one Method Blank. For biota analysis, leave the Method Blank empty. A Biota Control Blank is also prepared by weighing 0.6 g of homogenized biota control blank tissue into a digestion vial.

12.1.3.5 Prepare the LCS for biota samples by weighing 0.6 g of homogenized biota control blank into the digestion vial. Add 1.5 mL of Hg ICV Intermediate the vial. Only prep a Laboratory Control

Spike Duplicate (LCSD) if requested by client or there is not enough sample to run an MS/MSD. Prepare the LCSD in the same fashion as the LCS.

12.1.3.6 For biota samples, weigh approximately 0.05 g of Standard Reference Material into a labeled digestion vial. The analytical balance must read to at least 4 places past the decimal.

12.1.3.7 Weigh 0.6 g of homogenized sample into a labeled digestion vial. Record the weight to the nearest 0.01 g in the digestion prep log.

12.1.3.8 To prepare a Matrix Spike and Matrix Spike Duplicate, a sample with sufficient volume is chosen at random. Weigh 2 additional 0.6g aliquots into digestion vials. Add 1.5 mL of Hg ICV Intermediate to each vial.

12.1.3.9 Add 30 mL of 3% HNO3 solution to all QC and samples. Swirl digestion vials to gently mix.

12.1.3.10 To each digestion vial add 0.5 mL of concentrated nitric acid. Swirl digestion vials to gently mix.

12.1.3.11 To each digestion vial add 2.0 mL of concentrated hydrochloric acid. Swirl digestion vials to gently mix.

12.1.3.12 Add 6.0 mL of 5% KMnO4, place lid or cap on tube so as to allow pressure to vent but minimizing evaporation, swirl the digestion vials gently, and let stand for 15 minutes. If a sample does not maintain a purple or brown color, an additional 3.0 mL of KMnO4 solution is added to all samples, QC, and standards. **Note: If the sample still does not maintain color, discard set and dilute the sample prior to digestion.**

12.1.3.13 Digest samples for 30 minutes at 95+5°C.

12.1.3.14 Remove samples and let cool to room temperature.

12.1.3.15 Add 3.0 mL of 12% hydroxylamine hydrochloride solution. Swirl digestion vials to gently mix.

12.1.3.16 All samples and QC need to be diluted by a dilution factor of 5. Add 2.0 mL of sample to a digestion vessel. Dilute the sample by adding 8.0 mL of 7471 Blank water.

12.1.3.17 Samples are now ready for analysis.

12.2 Basic System Operation – Analytical

12.2.1 This portion of the SOP is designed to allow the user to set up and run a method, print a sample report, then shut down using the more basic software functions. For a more detailed explanation of the many other options, the user should refer to the Reference Manual.

12.2.1.1 Verify the carrier argon gas supply is on.

12.2.1.2 Switch on the CETAC M-7500.

12.2.1.3 Verify the computer is on. If not switch on the computer and enter network password and click on Start.

12.2.1.4 Go to programs, CETAC QuickTrace, and click on QuickTrace.

12.2.1.5 To create a new worksheet from a Template, Click on File, New From. Click on the browse button to select a Template worksheet, then enter the new File name (ex. Naming file 0713111 denotes the month, date, and year. The number at the end of the file is used to describe each run within a day's output). Click Save. Click ok.

12.2.1.6 To open an existing worksheet, select Open, and select the desired worksheet. The worksheet will open to the sequence page.

12.2.1.7 Make changes to the calibration curve Ids by clicking on Method Editor, Standards, Tools, Standard Names and change Ids in locations 1-6. To enter sample information, go back to the sequence page. Follow the Template to fill in Ids for the Calibration QC and samples, under the sample label column.

12.2.1.8 Go to File, Save to ensure changes to the worksheet are saved.

In the Method Editor Page the Conditions are set at:

Gas Flow (mL/min)	100
Pump Speed (%)	50
Sipper Depth (mm)	145
Sample Uptake (s)	35
Rinse Time (s)	80
Read Delay Time (s)	57
Replicate Read Time (s)	1.5
Replicates	4

12.2.1.9 Under Method editor, click QC Tests to Set the QC concentration and the control limits.

12.2.1.10 On the Sequence Parameters page hit the control button to set up when the QC will be analyzed, i.e. after calibration, after 10 samples, and at the end. IF re-calibration occurs the QC must be analyzed at the same frequency. This page also allows you to put the system into standby mode. The options allow you turn the pump on "slow" or "off. It also allows you to turn the Lamp off and Gas off.

12.2.1.11 The Sequence Parameters under the reports button allows you to customize the report printout by selecting solution information, report contents, and default number format.

12.2.1.12 Under the Sequence Editor page click on Manual QC, specify what to do if calibration QC fails, for example stop analysis, flag and continue, repeat-flag and continue, recalibrate and repeat, recalibrate and repeat with samples, or reslope and repeat.

12.2.1.13 On the Auto QC page of the Sequence Editor, specify what QC to run at the end of a run and corrective action if QC fails

12.2.1.14 Under the Sequence Editor click Sequence... This is where you determine the number of samples in the analysis and how often calibrations will be performed.

12.2.1.15 The analysis screen is accessed by clicking on the analysis button, from the main toolbar. This screen is where analysis controls and displays are located.

12.2.2 Starting the Analysis

12.2.2.1 Prepare the required reagents: Acid Rinse solution and reducing agent.

12.2.2.2 Turn on the lamp and carrier gas. A minimum 15 minute warm up time is required.

12.2.2.3 Put peristaltic tubing in place, and clamp in place.

12.2.2.4 Place the auto-sampler rinse tubing into the Acid Rinse bottle. If rinse pump is not on make sure it's on by clicking on the instrument page and clicking "pump on". Make sure to have probe down at this time as well.

12.2.2.5 Place the SnCl2 line in a bottle of reagent water and start the peristaltic pump. Inspect flow to make sure lines are flowing correctly and not pulsing.

12.2.2.6 Wet the GLS (Gas Liquid Separator) center post. In the software click on the instrument icon, click analyzer, set gas flow to 350-mL/min and change pump speed to 100%. Pinch the drain line until 2 or 3 bubbles go to the top of the GLS center post. Then release the drain line and allow liquid to restore itself.

12.2.2.7 Attach GLS exhaust tube to GLS center post and close the optical cabinet door. Place reagent capillaries in appropriate reagent bottles.

12.2.2.8 Open the appropriate worksheet and verify that the gas flow and pump speed in the worksheet matches what is listed in instrument/analyzer, if the flow and the speed is not the same make the necessary change or click the auto set icon on the menu bar. This will stabilize the instrument before auto-zeroing and running a peak profile.

12.2.2.9 Record the Lamps mA's in a daily instrument logbook by clicking on the instrument icon, clicking on analyzer, "status of lamp".

12.2.2.10 Peak profile the high standard of the calibration. To do this click on Method Editor, read a sample icon, and then choose the location of high standard. Record the concentration of the peak profile standard in a daily instrument logbook.

12.2.2.11 Hit the GO icon to start the calibration. Once the calibration is complete a dialog window will generate stating, "Continue with Analysis" Click YES if satisfied with Calibration or click NO to re-analyze Calibration.

12.2.2.12 Hit the STOP icon to immediately end an analysis that is currently running. The autosampler probe will immediately return to the rinse station. Stopping an analysis in progress will prevent data from being saved for that sample.

12.2.2.13 To stop the analysis after the current sample select the Stop/After solution item from the analyze menu.

Note: If you stop during sample uptake, or before the rinse has been completed, make sure you allow sufficient time in the rinse station before re-starting the analysis.

12.2.2.14 If you stopped the analysis, you can simply restart by clicking the GO button. The analysis will begin at the next un-analyzed solution within the sequence.

12.2.2.15 To restart the analysis from the beginning you will need to generate the sequence again. Open the Sequence Editor, make any desired changes, click on Generate Sequence. Then click GO to start analysis over.

12.2.2.16 To read a single sample, click on the icon, analyze single sample. A dialog window will be brought up where you will enter the tube position, sample label, sample type, and other information if desired.

Note: Using the read sample feature may invalidate your QC setup. If you have stopped during a batch analysis to read a single sample, it will be inserted into the sequence after the last analyzed sample.

12.2.2.17 When the run is complete the instrument will automatically go into standby mode. Depending what options you have selected from sequence parameters, this may turn off or slow the pump, turn off the lamp, and/or the carrier gas flow.

12.2.2.18 To export the data click on the sequence parameters icon. Make sure the "After run" is checked. In the file name section enter where you want the run to export i.e. V:\Metals\CVAA\40HG2\output. Then go to File, export, ok.

12.2.3 System Shutdown

12.2.3.1 Place the inlet of the SnCl2 line in a container of 10% nitric acid rinse (use a 125 mL container; use 12.5 mL of Nitric and dilute to 125 mL with reagent water) for 10 minutes. ..

12.2.3.2 Once the SnCl2 is rinsed with 10% nitric acid rinse, place the SnCl2 line into DI water for 1 minute to rinse system.

12.2.3.3 Remove the SnCl2 line from the DI water and raise lines out of rinse solution. Run the pump until the lines are dry.

12.2.3.4 Raise the probe from the rinse station by clicking on the instrument icon and by clicking the Move Sipper Up button. Turn off peristaltic pump by clicking the Pump Off button.

12.2.3.5 Release all four peristaltic pump channel clamps and remove the tubing from the channel.

12.2.3.6 Remove the GLS exhaust tube from the GLS center post.

12.2.3.7 Turn off the gas and lamp by clicking on the instrument icon, then click analyzer. Turn the Lamp Off and set the gas to 0.

11.2.3.8 Leave the instrument on until you next use it. Close out of the software.

13. QUALITY CONTROL

13.1 Calibration Blanks – Calibration blanks (ICB and CCB) may not contain concentrations in excess of the Pace Reporting Limit. If blank results are not acceptable, analysis of the batch should be halted. Corrective actions could include preparation of fresh calibration standards and blanks.

13.2 Calibration Checks- Calibration checks (ICV and CCV) must agree within 10% of the true value. If the results are unacceptable, re-calibrate the system and then re-analyze the check solution and samples.

13.3 Pace Reporting Limit Standard (PRL or CRDL Std) – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration, recovery 60-140% of true value. When analyzing by 7471B, CRDLs must, at a minimum, bracket all samples and have a recovery of 70-130% of the true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.4 Laboratory Control Spike – A Laboratory Control Spike (LCS) must be prepared and analyzed with every sample batch or every 20 samples, whichever is more frequent. A Laboratory Control Spike Duplicate (LCSD) is performed if there is insufficient sample available for a MS/MSD or if requested by the client. The LCS and LCSD must meet criteria 85-115% recovery or all the samples prepared in the batch must be re-prepped and re-analyzed. The acceptance criterion for precision is 20% RPD. If insufficient sample remains to re-prep and re-analyze, the data qualifiers is given to all associated samples.

13.5 Method Blank – A Method Blank (MB) must be prepared and analyzed with every sample batch or every 20 samples, whichever is more frequent. The MB must not contain mercury at a concentration at or above the Pace Reporting Limit. Any samples digested with an unacceptable method blank must be re-prepped and analyzed unless the sample concentrations are less than the reporting limit or more than 10 times the value found in the method blank. In those cases that MDL reporting is required, the MB must be evaluated to the MDL. For MDL reporting, an appropriate data qualifier is given to samples associated with +/-MB hits between the +/-MDL and +/-PRL where the sample results are less than 10 times the value found in the method blank. Please refer to the attachment section for the flagging charts.

13.6 Matrix Spike/ Matrix Spike Duplicate – A Matrix Spike/Matrix Spike Duplicate (MS/MSD) must be prepared and analyzed for each batch or every 20 samples that are similar in matrix at a frequency of 5%. For TCLP, SPLP, and ASTM samples one Matrix Spike (MS) must be prepared per sample matrix (soil and wood chips would each require a separate matrix spike). The sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD. If one or both spike recoveries are outside 85-115% recovery acceptance criteria, the parent sample is given an appropriate data qualifier. If the RPD is outside 20% RPD criteria the parent sample is given an appropriate data qualifier. Parent sample is chosen at random or assigned by the client.

13.7 All reported results must be within the range of the calibration curve. Dilute when results are greater than the high standard in the curve.

14. DATA ANALYSIS AND CALCULATIONS

14.1 **Water/TCLP/SPLP/ASTM Samples** – Since initial sample aliquot and final digestate volumes are the same, the mercury analyzer data system will calculate the concentration directly. No further calculations are necessary unless the sample was diluted.

14.2 Soil/Solid/Biota Samples -

Final Result (mg/kg dry weight corrected) =

$$\frac{\text{Raw data result (µg/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{Sample weight (g)} * \text{Dry weight for soil/solid (decimal form)}}$$

Biota results are reported on an as is/wet weight basis. The dry weight correction in the formula is not applicable.

14.3 Wipes

Final Result (Total µg) =

$$\frac{\text{Raw data result (µg/L)} * \text{Final Volume (L)} * \text{Dilution Factor}}{\text{1 wipe}}$$

14.4 Accuracy-

$$\text{Spike Percent Recovery} = \frac{\text{Spike Sample Result} - \text{Sample Result}}{\text{Spike Added}} \times 100$$

14.5 Precision-

$$\text{Relative Percent Difference} = \frac{\text{Spike Sample Result} - \text{Spike Sample Duplicate Result}}{(\text{Spike Sample Result} + \text{Spike Sample Duplicate Result})/2} \times 100$$

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

Table 5
QC SUMMARY

Analytical Method ⇨ Calibration Measure ⇩	**EPA SW846 7470, 7471, 7471M for Tissue** **Frequency**	**Acceptance Criteria**
Laboratory Control Spike and Laboratory Control Spike Duplicate	• One LCS per batch of samples, up to 20 environmental samples, whichever is more frequent. • A LCSD is required if MS/MSD is not performed or if requested by the client.	• Project Specific or • For 7470 and 7471 85 –115% with 20% RPD • For Biota by 7471 70-110% with 10%RPD
Matrix Spike / Matrix Spike Duplicate (MS/MSD)	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	• Project Specific or • For 7470 and 7471 85 –115% with 20% RPD • For Biota by 7471 70-110% with 10%RPD
Method Blank	• One per batch of samples, up to 20 environmental samples, whichever is more frequent.	• Project Specific or • Less than the RL (LOWEST STANDARD IN CURVE)
Initial Calibration	• Analyzed daily before samples • Minimum 5 standards plus a blank	• Correlation coefficient must be 0.995 or greater
Initial Calibration Verification (ICV)	• Analyzed after calibration.	• Recovery must be between 90 – 110%
Initial Calibration Blank (ICB)	• Analyzed after ICV.	• Project specific or • Less than RL (LOWEST STANDARD IN CURVE)
CRDL	• 7470, 7471A, 7471M Analyzed after Initial calibration blank. • 7471B At a minimum, CRDLs must bracket all samples reported by this method.	• 7470, 7471A, 7471M 60-140% • 7471B 70-130%
Continuing Calibration Verification (CCV)	• Analyzed after every 10 samples.	• Project specific or • Recovery between 90 – 110%
Continuing Calibration Blank (CCB)	• After each CCV.	• Project specific or • Less than RL (LOWEST STANDARD IN CURVE)

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

Table 6
DATA ASSESSMENT/CORRECTIVE ACTION

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation: EPA SW846 7470, 7471, 7471M for Tissue** **If these conditions are not achieved ⇒**
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
Initial / Continuing Calibration Verification	• 5
Initial / Continuing Calibration Blank	• 6
Holding Time Compliance	• 7
CRDL	• 8

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 times the method blank contamination are reported without qualification.

2. In the absence of project specific or method requirements, in-house generated limits will be used. If the MS or MSD fail because the concentration of the spike is less than 25% of the concentration of the parent, use appropriate flag for the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, use appropriate flag for the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate precision data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and/or appropriate precision qualifier to reflect the deficiencies. Generate a Non-Conformance Memo.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed.

6. If ICB/CCB is outside the control limits, reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB do not have to be reanalyzed or qualified.

7. Flag results with the appropriate data qualifier.

8. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 See Section 16, Table 6: Corrective Action.

18. METHOD PERFORMANCE

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedure*s. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

18.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of the LOD and LOQ*, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

18.1.4 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program*, to demonstrate continuing competence. All results are stored in the QA office.

19. METHOD MODIFICATIONS

19.1 For SW846 7470A, the lab has chosen to use a 10mL sample volume instead of the method specified 100mL. All standards and reagents have been reduced to ensure that the ratios are still consistent with the method.

19.2 For SW846 7471A/B, the lab has chosen to use a 0.3g sample volume instead of the method specified 0.5-0.6g. All standards and reagents have been reduced to ensure that the ratios are still consistent with the method.

19.3 SW846 7471A/B has been modified to use in the digestion and analysis of biological tissue samples. Sample volume, reagents and standards have not been altered.

20. INSTRUMENT/EQUIPMENT MAINTENANCE

20.1 Any daily or periodic maintenance must be recorded in the instrument daily logbook.

21. TROUBLESHOOTING

21.1 Please see the instrument manual for information on instrument troubleshooting.

22. SAFETY

22.1 Standards and Reagents - The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses.

22.2 Safety Data Sheets (SDSs) – A reference file of SDS are on file in the laboratory and available to all personnel. A formal safety plan has been prepared and distributed to all personnel with documented training.

22.3 Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion. All digestions must be conducted in a properly functioning fume hood.

22.4 Samples - Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.
Note: Extreme caution must be used when preparing rodents for digestion. The samples must undergo a special procedure to destroy any Hantavirus, which may be present. Refer to the most recent version of SOP S-GB-L-002 *Small Rodent Handling and Homogenization* for details.

22.5 Analysts should take necessary safety precautions when handling chemicals and samples. Proper personal protective equipment may include safety gloves, lab coats, and safety glasses or goggles. Analysts should be familiar with the SDS sheets for all chemicals and reagents they use for this procedure and the location of the SDS sheets within the laboratory. Any questions or concerns should be taken to the laboratory Chemical Hygiene/Safety Officer.

23. WASTE MANAGEMENT

23.1 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-W-001, *Waste Handling and Management*.

24. POLLUTION PREVENTION

24.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

25. REFERENCES

25.1 SW-846 7470A, Revision 1, September 1994

25.2 SW-846 7471A, Revision 1, September 1994

25.3 SW-846 7471B, Revision 2, February 2007

25.4 PASI Quality Manual, current revision

25.5 National Environmental Laboratory Accreditation Conference (NELAC), July 2003 Standards.

25.6 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version

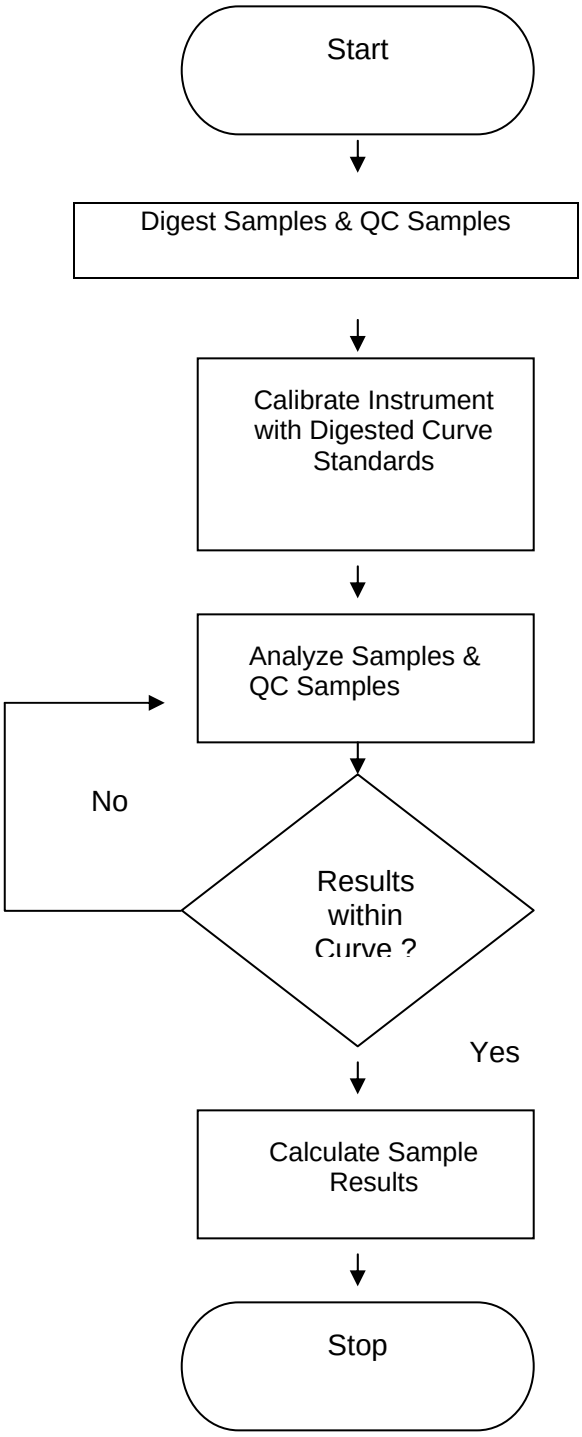
26. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

26.1 Mercury Process Flow Chart

27. REVISIONS

Document Number	Reason for Change	Date
S-GB-M-017-Rev.0	First Issue	03Aug2011
S-GB-M-017-Rev.01	General: Updated SOP to new format Section 9: Added Hot block and DORM-4 SRM. Removed water bath and DOLT-3 SRM. Section 13.3: Added 7471B CRDL frequency and criteria of 70-130%.	08Apr2013

Mercury Process Flow Chart



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STANDARD OPERATING PROCEDURE

DETERMINATION OF HEXANE EXTRACTABLE MATERIAL (HEM) AND SILICA GEL TREATED-HEXANE EXTRACTABLE MATERIAL (SGT-HEM) IN SOLIDS

Reference Methods: EPA 9071B

Local SOP Number:	S-MN-I-357-rev.20
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-357-rev.19

APPROVALS

Laboratory Assistant General Manager — Date: 3/5/2014

Laboratory Quality Manager — Date: 06Mar2014

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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1. Purpose/Identification of Method

1.1. This Standard Operating Procedure provides detailed instructions for the gravimetric determination of n-hexane extractable material (HEM; oil and grease) and silica gel-treated n-hexane extractable material (SGT-HEM; total petroleum hydrocarbon) in solid samples as delineated in EPA Method 9071B.

2. Summary of Method

2.1. A nominal 20-gram aliquot of soil or other solid matrix sample is homogenized within its respective container and subsequently added to a soxhlet thimble containing 20 grams of sodium sulfate. Approximately 100 milliliters of n-hexane is cycled through the sample continuously over the course of four hours after which it is poured off into a pre-weighed tin, evaporated and any remaining HEM residue is measured gravimetrically.

2.2. For SGT-HEM determination the residue is re-dissolved in hexane and an amount of silica gel is added to the solution to remove polar materials. The solution is filtered, evaporated, desiccated, and weighed.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameter**s: Extractable materials that may be determined are relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related materials. The term “n-hexane extractable material” reflects that this method can be used to determine materials other than oils and greases such as sulfur compounds, organic dyes and chlorophyll.

3.3. This procedure is not applicable to measurement of materials that volatilize at temperatures below 85ºC. Petroleum fuels from gasoline through #2 fuel oil may be partially lost during the solvent removal process. In addition, some crude oils and heavy fuel oils may contain materials that are not soluble in n-hexane, thus recoveries of these materials may be low.

4. Applicable Matrices

4.1. This SOP is applicable to solid samples.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes is 250 mg/kg for this method. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Interferences extracted from samples will vary considerably from source to source, depending upon the nature of the site being sampled. A smaller sample volume may be needed for extraction for samples which consist of complex matrices containing substances such as particulates or detergents that may interfere with the extraction procedure.

6.2. This SOP is applicable to samples containing HEM in concentrations from 250-50000 mg/L. If clients consistently send samples with residue that exceeds the upper detection limits of 50000 mg/L, reduced volumes must be used to allow for results within the detection range of 250 to 50000 mg/L of residue.

6.3. Hexane has the ability to dissolve not only oil and grease but also other organic substances. No known solvent will dissolve selectively only oil & grease. Solvent removal results in the loss of short-chain hydrocarbons and simple aromatics by volatilization.

6.4. The filter paper may have the potential to inflate results and must be rinsed with hexane prior to use.

6.5. Silica gel has the potential to inflate results for SGT-HEM if it is transferred with the hexane into the pan for gravimetric determination.

6.6. All solvents must have an evaporated residue of < 1.0 mg HEM/L.

6.7. Samples with high sulfur content may interfere with results due to the solubility of sulfur in hexane.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	Samples are collected and stored in wide-mouth glass jars following conventional samples practices	None	Above freezing but below 6°C	28 days of collection

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Aluminum dishes	150 mL, smooth-sided	Fisher Scientific # 08-732-104, or equivalent
Stainless steel funnels	Used to filter out solids in extract, if necessary	Fisher Scientific or equivalent
Glass fiber filter paper	Used to filter out solids in extract, if necessary. Manufacturer by Whatman	Fisher Scientific # 09-801E, or equivalent
Soxhlet extractors, condensers and 250-mL Erlenmeyer flasks	Used to extract HEM from solids	Fisher Scientific or equivalent
Multi-Unit Extraction Heater	Used to heat hexane for extraction of soils	Fisher Scientific or equivalent
Cellulose extraction thimbles	33mm x 94mm. Manufactured by Whatman	Fisher Scientific # 09-924-232, or equivalent
Analytical balance	Capable of weighing 0.1 mg	Fisher Scientific or equivalent
Stir plate and magnetic stir bars	Used in SGT-HEM extraction	Fisher Scientific or equivalent
Desiccator with Drierite	Used in final drying process	Fisher Scientific or equivalent
Microsoft Excel	Spreadsheet software	See master list for current version
EPIC Horizon	Data Reporting Software	See master list for current version

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
Hexadecane/Stearic Acid Standard	Snip and pour standard. 40 mg HEM total, 20 mg SGT-HEM	Environmental Express part # G3025
n-Hexane	< 1 mg HEM/L residue. Store at room temperature. Expires one year after opening.	Fisher part # H303-4 or equivalent
Sodium Sulfate (Na_2SO_4)	Anhydrous granular, 10-60 mesh.	Fisher part # S415-200LB
Silica Gel	Anhydrous, 50-150 µm mesh. NOTE: 75-150 mesh is not available.	Fisher part # 04669-1 or equivalent
Drierite	Store at room temperature in a tightly closed glass jar.	Fisher part # 075773B Blue indicator: part # 075783B

10.1.1. Sodium Sulfate: Bake for 4 hours at 400ºC. Store in a tightly closed glass container at room temperature.

10.1.2. Silica Gel: Bake for at least 24 hours at 200-250ºC. Store in a tightly closed glass container at room temperature.

10.1.3. Drierite: Use a mix of white and blue crystals in a closed desiccator. When the blue crystals begin to turn purple or pink, the Drierite is losing its effectiveness and must be replaced or regenerated. See S-MN-O-557 (or equivalent replacement) for drierite regeneration procedure.

11. Calibration and Standardization

11.1. Daily calibration of the balance is required following SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Record in associated balance calibration logbook. Calibration limits are found in the balance calibration logbook; if values exceed these limits, recalibrate the balance.

11.2. All balances must be certified by an outside agency on an annual basis with documentation of the calibration maintained in the QA office.

11.3. Calibration of the balance must be verified before and after each analytical batch.

12. Procedure

12.1. Aluminum Dish Preparation

12.1.1. Label one aluminum dish for each sample and QC sample.

12.1.2. Using the analytical balance, obtain a tare weight for each dish and record it on the bench sheet (see Attachment I).

12.2. Determination of HEM by Soxhlet Extraction

12.2.1. Weigh 20 g $Na_2SO_4$and add to a clean extraction thimble. Tare the thimble and Na_2SO_4 then add 20 g of homogenized sample.

12.2.1.1. Reduce the amount of sample added to the thimble if the matrix appears to contain or smells of fuel oil, tar or other oily organic matter. In such cases, the amount used for analysis is determined by the best judgment of the analyst and in the event of an elevated non-detect, may require re-preparation of the sample at a larger volume.

12.2.1.2. If the sample contains a large amount of water, the volume must be reduced. This is due to the Na_2SO_4 absorbing the water, hardening and not allowing the hexane to cycle through the sample properly, thus biasing the results low.

12.2.1.3. For the method blank (MB) and laboratory control sample (LCS), no additional sample is added to the thimble other than the 20 g of Na_2SO_4. These QC samples are otherwise processed in the same manner as the rest of the batch.

12.2.2. Assemble the soxhlet extraction vessel and add 100-125 mL of hexane to the vessel, allowing it to drain into the flask.

12.2.3. Place the thimble containing the sample into the soxhlet extraction vessel.

12.2.3.1. Add the contents of one tube of hexadecane/stearic acid standard to each thimble for the LCS and MS/MSD.

12.2.4. Place the extraction vessel into the heating blocks and connect the water condenser. Start the water chiller to allow cool water to circulate through the condensers and turn the heating mantels on.

12.2.5. Once the hexane has come to a boil, allow the samples to extract for 4 hours. After 4 hours, turn off the heating mantels and allow the samples to cool for 30 minutes, while water is still circulating. Remove the vessels from the heating blocks and drain all the hexane form the extraction thimble into the flask. Discard the thimble and detach the flasks from the soxhlet vessel.

12.2.6. Decant the hexane extract into a pre-weighed aluminum dish labeled with the sample ID. If the hexane extract contains solids or water, pour it through a funnel and glass fiber filter paper that

have been pre-rinsed with hexane. Rinse the flask three times with hexane to ensure the entire sample has been transferred to the dish.

12.2.7. Place the dishes on a hotplate set at 40°C in a fume hood to evaporate the solvent. When evaporation is complete, transfer the dishes to a desiccator to allow equilibration, preferably overnight. Proceed to section 12.3.

12.3. Gravimetric Determination of Results

12.3.1. Weigh each dish to 0.1 mg and record the weight on the bench sheet (see Attachment I). Return the dish to the desiccator after recording the weight.

12.3.2. After at least one hour, weigh each dish again and record. If second weight is within ±0.5 mg of the first, constant weight is established and analysis is complete. If it is not within ±0.5 mg, return the dish to the desiccator and repeat this step for a third weight and, if necessary, a fourth weight.

12.3.2.1. If the third weight measurement agrees within 4% of the first weight, it is considered a constant weight and analysis is complete.

12.3.2.2. If the fourth weight is still not within ±0.5 mg, use the fourth weight and qualify the sample data as not having established constant weight.

12.4. Determination of SGT-HEM

12.4.1. Follow the procedure as outlined in section 12.2 or 12.3.

12.4.2. Re-dissolve the HEM residue in approximately 20-30 mL of hexane and transfer to a large fluted aluminum dish. Rinse the dish thoroughly with hexane in order to collect any residue present in the dish.

12.4.3. Add ~3 g silica gel for every 100 mg of HEM residue. Add a stir bar.

12.4.4. Stir for ~5 minutes then sit for ~1 minute to allow silica gel to settle. Pour through hexane-rinsed phase separator filter paper collecting it in the same aluminum dish used for HEM determination.

12.4.5. Place the dish on a hot plate set at 40ºC in a fume hood to evaporate the solvent. When evaporation is complete, transfer the dish to a desiccator to allow equilibration, preferably overnight.

12.4.6. Weigh to a constant weight as in section 12.3.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Na_2SO_4	Once per batch of up to 20 samples.	Must contain less than 250 mg/kg HEM or SGT-HEM.	If the method blank does not meet this criterion, the source of the contamination must be investigated and documented, and attempts made to eliminate the source. Reanalyze all samples associated with the failed method blank. If sufficient sample volume is not available to reanalyze samples, report the sample data with a qualifier on each sample associated with the non-compliant method blank. ***Exceptions:*** If sample results are non-detect, report results without qualification. If sample results are 10x or greater than the MB result, report results and narrate on the validation report as not impacted by the blank

				contamination. If sample results are less than 10x the MB result and the sample cannot be reanalyzed, report the results with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	Na_2SO_4 with one tube of hexadecane / stearic acid standard added	Once per batch of up to 20 samples. If there is insufficient sample volume for matrix spike and duplicate analysis, an LCSD must be performed.	HEM recovery must be within 78-114% of the 2000 mg/kg true value. SGT-HEM recovery must be within 64-132% of the 1000 mg/kg true value. RPD requirements of the LCS and LCSD: HEM $\leq$ 18% SGT-HEM $\leq$ 34%.	If the recovery is not within control limits, check for errors in calculations, standards preparation and spiking of the LCS solution. If an error or problem is found and can be corrected by amending the calculations and the result falls within the limits, accept the data and report without a qualifier flag. If no errors are found, re-prep and re-analyze the LCS as long as re-analysis occurs on the same day as the original analysis. If re-analyzed LCS fails, re-analyze the all samples associated with the failed LCS. If sufficient sample volume is not available to reanalyze samples, report the sample data with a qualifier on each sample associated with the non-compliant LCS. ***Exceptions:*** If the LCS recovery is greater than the acceptance criteria and the sample results are non-detect, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	Client-provided sample with one tube of hexadecane / stearic acid standard added.	Once per batch if sufficient volume of sample is available. An MSD can be performed per client request and may take the place of a sample duplicate (DUP).	HEM recovery must be within 78-114% of the 2000 mg/kg true value. SGT-HEM recovery must be within 64-132% of the 1000 mg/kg true value. RPD requirements of the MS and MSD: HEM $\leq$ 18% SGT-HEM $\leq$ 34%.	If the recovery is not within control limits or the RPD exceeds control limits in the case of an MS/MSD, check for errors in calculations, standards preparation and spiking of the MS solution. If an error or problem is found and can be corrected by amending the calculations and the result falls within the limits, accept the data and report without a qualifier flag. If no errors are found, report the sample data with a qualifier on the MS and parent sample. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.
Duplicate Sample (DUP)	Client-provided sample.	Once per batch if sufficient volume of sample is available. An MSD may be performed in place of a DUP.	RPD requirements of the parent sample DUP: HEM $\leq$ 18% SGT-HEM $\leq$ 34%.	If the RPD exceeds control limits, check for errors in calculations and sample preparation. If an error or problem is found and it can be corrected by amending the calculations and the result falls within the limits, accept the data and report without a flag. If no errors are found, report the sample data with a qualifier on the DUP and parent sample.

14. Data Analysis and Calculations

14.1. HEM or SGT-HEM Recovery

14.1.1. To determine the mg/kg results for soils on a wet weight basis:

$$\text{HEM or SGT - HEM, mg/kg} = \frac{(\text{F - I})}{W} \times \frac{1000\,\text{g}}{kg}$$

Where: F = Final dish weight, mg
I = Initial dish weight, mg
W = Weight of sample, g

14.1.2. To determine soils based on a dry weight of the result calculated in 14.1.1, see SOP S-MN-I-367 Percent Moisture, or equivalent replacement:

$$\text{HEM or SGT - HEM (Dry), mg/kg} = \frac{W}{100 - M} \times 100$$

Where:
W = FWet weight result, mg/kg from 14.1.1
M = Percent moisture

14.2. LCS and MS/MSD % Recovery

$$\%\,\text{Recovery} = \frac{SS - PS}{SA} \times 100$$

Where:
SS = Spiked Sample result, mg/kg
PS = Parent Sample result, mg/kg (only applicable to MS/MSD, LCS = 0)
SA = Spike Added from spiking standard, mg/kg

14.3. Relative Percent Difference (RPD)

$$RPD = \frac{|PS - Dup|}{(PS + Dup)/2} \times 100$$

Where:

PS = Parent Sample result
Dup = Duplicate sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 & 13.

16. Corrective Actions for Out-of-Control Data

16.1. See tables in section 11 & 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the tables in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Demonstration of Capability (DOC):** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. Evaporation of the solvent at 40ºC on a hot plate has been demonstrated to be an effective method modification according to SOP S-MN-Q-269. See sections 12.2.7 and 12.4.5.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

22.3. Extraction of samples with hexane should be performed in a hood with the analyst wearing safety glasses and latex gloves. Inhalation of high concentrations can produce asphyxiation, while prolonged skin contact may cause defatting and possible dermatitis. Hexane is flammable and should be used with caution.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. EPA Method 9071B: Oil and Grease Extraction Method for Sludge and Sediment Samples.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I – Oil and Grease Bench sheet (example)

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-357-rev.19	Implemented new corp SOP format Removed 1664A and changed 9071A to 9071B throughout 2 – updated for 9071B 3.2 – updated 3.3 – added 4.1 – removed aqueous 6.1, 6.2, 6.4-6.7 – added 6.8 – removed 7.1 – removed aqueous 7.2 – removed 9.1 – updated all, removed equip for aqueous samples 10.1 – added spiking standard, removed LCS soln and food coloring 12 – changed all "tin" to "dish", removed all aqueous prep 12.2.1.1-12.2.1.3. – added 12.2.3.1. – added 12.2.5. – added "once the hexane comes to a boil" and "discard the thimble…" 12.2.6. – added hexane procedures 12.2.7. – added "when evaporation is complete…" 12.3. – added 12.4.1. – 12.4.5. – added 14.2. – added 14.3. – added 25.5, 25.7 – removed Attachment I – updated	10May2013
S-MN-I-357-rev.20	Updated items in table 10.1 Added 10.1.1-10.1.3 Added 11.3 – calibration of balance 12.2.3.1 – removed spiking and added hexadecane Updated table 13.1 Removed DoD reference in section 25 Attachment I updated	28Feb2014

ATTACHMENT I – Oil and Grease Benchsheet (example)

Pace Analytical	Document Name: Oil and Grease Bench Sheet	Date Revised: 23Jul2013
		Page 1 of 1
	Document Number: F-MN-L-086-Rev.21	Issuing Authority: Pace Minnesota Quality Office

Method: Click to Select
Extraction Method: Click to Select
Type: Click to Select

Batch:
Instrument ID: Click to Select

Extraction Date:
Analyst:
Reviewed:

Sample	Results (mg/L) (mg/kg)	Raw mg	Pan ID	Sample Aliquot (mL)	pH	Extraction Begin time	Extraction End time	Initial Weight (g)	First Weight (g)	Second Weight (g)	Third Weight (g)	Fourth Weight (g)	Final Weight (g)
Weighing Information: Date/Time/Analyst:													
MB	#DIV/0!	0.0	MB										0
LCS	#DIV/0!	0.0	LCS										0
LCSD or MS	#DIV/0!	0.0											0
MSD or DUP	#DIV/0!	0.0											0
	#DIV/0!	0.0	1										0
	#DIV/0!	0.0	2										0
	#DIV/0!	0.0	3										0
	#DIV/0!	0.0	4										0
	#DIV/0!	0.0	5										0
	#DIV/0!	0.0	6										0
	#DIV/0!	0.0	7										0
	#DIV/0!	0.0	8										0
	#DIV/0!	0.0	9										0
	#DIV/0!	0.0	10										0
	#DIV/0!	0.0	11										0
	#DIV/0!	0.0	12										0
	#DIV/0!	0.0	13										0
	#DIV/0!	0.0	14										0
	#DIV/0!	0.0	15										0
	#DIV/0!	0.0	16										0
	#DIV/0!	0.0	17										0
	#DIV/0!	0.0	18										0
	#DIV/0!	0.0	19										0
	#DIV/0!	0.0	20										0

Comments:

Oil & Grease (mg/L) = (Final Wt.(g)) - (Initial Wt.(g)) / (Volume (mL)) x 1000000 Oil & Grease (mg/Kg) = (Final Wt. (g)) - (Initial Wt. (g)) / (Weight (g)) x 1000000

Reagents	Vendor	Pace ID #	Reagents	Vendor	Pace ID #
Hexane	Fisher		Extraction Disk	Environ. Express	
LCS/MS spike soln	Environ. Express		Soxhlet Thimbles	Fisher	
Na2SO4	Fisher		Acetone	Fisher	
Silica Gel	Fisher		Methanol	Fisher	
H2SO4/HCl	Fisher				

Balance Calibration		
Balance ID: 10406293		
Weight	Before	After
1000 mg ± 5 mg		
2.0 mg ± 0.2 mg		

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STANDARD OPERATING PROCEDURE

DETERMINATION OF TOTAL RECOVERABLE PHENOLICS BY KONELAB

Reference Methods: EPA 420.4

Local SOP Number:	S-MN-I-494-rev.13
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-494-rev.12

APPROVALS

Laboratory Assistant General Manager — Date: 5/3/2014

Laboratory Quality Manager — Date: 08May2014

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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1. Purpose/Identification of Method

1.1. This Standard Operating Procedure provides a detailed description of determining total phenol in environmental samples as delineated in EPA Method 420.4.

2. Summary of Method

2.1. The method is based on the distillation of phenol and subsequent reaction of the distillate with alkaline ferricyanide and 4-aminoantipyrine to form a red complex, which is, measured at 520nm.

2.2. Color response of phenolic materials with 4-aminoantipyrene is not the same for all compounds. Because phenolic type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason, phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.

2.3. Reduced volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method.

2.4. Limited performance based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in Quality Control Section.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameter**s: This SOP is capable of measuring phenolic materials from 0.05 to 1.0 mg phenol/L in the aqueous phase using phenol as a standard. Samples of greater concentration are determined by dilution into this range.

4. Applicable Matrices

4.1. This SOP is applicable to the analysis of drinking, ground, surface and saline waters, and domestic and industrial wastes.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes is 0.05 to 1.0 mg/L phenol for this method. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Acidifying the sample to a pH of 4.0 and aerating briefly by stirring eliminates interference from sulfur compounds.

6.2. Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate. If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

6.3. Background contamination from plastic tubing and sample containers is eliminated by avoiding the use of plastic sample containers and plastic tubing.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Samples are collected in glass bottles. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis and minimize waste disposal.	Samples must be preserved with sulfuric acid to pH < 2 and refrigerated.	Sample are stored above freezing but below 6°C	Analyzed as soon as possible after collection. If storage is required, samples maybe held up to a maximum of 28 days with suggested preservation and storage.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Volumetric flasks	Various, Class A	Fisher Scientific or equivalent
Volumetric pipettes	Various, Class A	Fisher Scientific or equivalent
Calibrated syringes	100 µL – 2.5 mL	Fisher Scientific or equivalent
Plastic digestion tubes	50 mL	Environmental Express # SC475
Konelab 20	Konelab 20 or equivalent	Fisher Scientific or equivalent
Hengar Boiling Stone		Fisher Scientific or equivalent
Distillation apparatus	Midi-Dist glassware or equivalent	
Miscellaneous laboratory glassware/ eqiuipment		
LIMSLink	Data transfer software	See master list for current version
Horizon, or equivalent replacement	Data reporting software	See master list for current version

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized water (DI)		Verify daily that pH and specific conductivity are within acceptable limits
4-Aminoantipyrine	Crystalline	Fisher part # O1123-100
Potassium Ferricyanide [$K_3Fe(CN)_6$]	Crystalline	Fisher part # P232-500
Boric Acid (H_3BO_3)	Powder	Fisher part # A74-500
Potassium Chloride (KCl)	Crystalline	Fisher part # P330-500
Curve Stock Standard	1000 mg/L. Store at room temperature. Expires per manufacturer's specifications.	Fisher part # 5740-4
QC Stock Standard	1000 mg/L. Store at room temperature. Expires per manufacturer's specifications.	ERA part # 982
Sodium Hydroxide (NaOH)	Pellets	Fisher part # S318-500

10.2. Table 10.2 - Working Solutions & Reagents

Solution / Reagent	Reagent(s) Used	Reagent(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Intermediate CurveStandard	Curve Stock Standard	0.1 mL	DI Water	9.9 mL	10 mL	10.0 mg/L
1.0 mg/L Curve Standard	Intermediate Curve Standard	1.0 mL	DI Water	9.0 mL	10 mL	1.0 mg/L
Intermediate QC Standard	QC Stock Standard	0.1 mL	DI Water	9.9 mL	10 mL	10.0 mg/L
ICV/CCV/LCS Solution	Intermediate QC Standard	0.5 mL	DI Water	9.5 mL	10 mL	0.5 mg/L
Color Reagent	4-Aminoantipyrine	0.16 g	DI Water	~250 mL	250 mL	N/A
Potassium Ferricyanide Solution	Potassium Ferricyanide	0.2 g	DI Water	~95 mL	100 mL	N/A
	Boric Acid	0.31 g				
	Potassium Chloride	0.375 g				
	1N NaOH Solution	4.7 mL				
1N NaOH Solution	NaOH Pellets	40 g	DI Water	~1 L	1 L	1 N

10.2.1. Intermediate Curve Standard – Add 0.1 mL Curve Stock Standard to ~5 mL DI water in a class A 10 mL volumetric flask. Dilute to volume with DI water. Store at room temperature. Prepare fresh daily.

10.2.2. 1.0 mg/L Curve Standard – Add 1.0 mL Intermediate Curve Standard to ~5 mL DI water in a class A 10 mL volumetric flask. Dilute to volume with DI water. Store at room temperature. Prepare fresh daily.

10.2.3. Intermediate QC Standard – Add 0.1 mL QC Stock Standard to ~5mL DI water in a class A volumetric flask. Dilute to volume with DI water. Store at room temperature. Prepare fresh daily.

10.2.4. ICV/CCV/LCS Solution – Add 0.5 mL Intermediate QC Standard to ~5 mL DI water in a class A 10 mL volumetric flask. Dilute to volume with DI water. Store at room temperature. Prepare fresh daily.

10.2.5. 4-Aminoantipyrine Color Reagent – Add 0.16 g 4-aminoantipyrine to ~150 mL DI water in a 250 mL volumetric flask and mix until dissolved. Dilute to volume with DI water. Store in a glass container at room temperature. Prepare fresh daily.

10.2.6. Potassium Ferricyanide Solution – Add 0.2 g potassium ferricyanide, 0.31 g boric acid and 0.375g potassium chloride to ~80 mL DI water in a 100 mL volumetric flask. Mix until dissolved. Add 4.7 mL 1N NaOH solution and dilute to volume. Store in a glass container at room temperature. Prepare fresh weekly.

10.2.7. 1N NaOH Solution – Add 40 g NaOH pellets to ~500 mL DI water in a 1 L volumetric flask and mix until dissolved. Allow solution to cool and dilute to volume with DI water. Store in a glass container at room temperature. Expires in six months.

11. Calibration and Standardization

11.1. Table 11.1 – Calibration and Standardization

Calibration Metric	Parameter/Frequency	Criteria	Comments
Calibration Curve Fit	Linear Regression	$r \geq 0.995$	If not met, remake standards and recalibrate and verify before sample analysis.

Initial & Continuing Calibration Verification (ICV/CCV)	An ICV must be analyzed immediately after each initial calibration. Prepare an ICV/CCV standard from a different source than the calibration standards. A CCV standard must be analyzed and reported at a frequency of 10% to ensure calibration accuracy during the analytical run.	90-110% of the true value	When measurements exceed the acceptance criteria, all analytical samples since the last compliant CCV must be reanalyzed. If the ICV or two consecutive CCVs exceed the control limits, the analysis must be terminated, the problem corrected and the system recalibrated.
Initial & Continuing Calibration Blank (ICB/CCB)	A calibration blank is prepared by adding all color development reagents to DI water and must be analyzed immediately after every ICV and the CCV.	Absolute value of the recovery must be less than the reporting limit. Per QAPP or client specifications, alternate criteria such as evaluating to ½ RL may apply.	If there is detection, stop analysis and determine the source of the contamination. Perform any necessary maintenance and recalibrate the instrument accordingly. Reanalyze any samples impacted since the last passing CCB. If two consecutive CCBs exceed the control limits, the analysis must be terminated, the problem corrected and the system recalibrated. ***Exception***: If all the affected samples are non-detect or at least 10x the concentration of the failing result, the sample data may be accepted.

11.2. Calibrate the instrument prior to use every day according to the Konelab SOP S-MN-I-507 (or equivalent replacement).A correlation coefficient of at least 0.995 is required in order to proceed with analysis.

11.2.1. The instrument will automatically prepare each standard in the calibration curve from the 1.0 mg/L calibration standard.

12. Procedure

12.1. Distillation

12.1.1. Adjust sample pH to ~ 4 by adding 1N NaOH solution drop-wise.

12.1.2. Measure 50 mL of sample (or an aliquot diluted to 50 mL with DI water) into a 50 mL reaction flask.

12.1.3. Add 3-5 Hengar boiling stones to the reaction flask.

12.1.4. Distill the samples using a midi-distillation apparatus (see Attachment I) until 45 mL has been collected. Stop the distillation; allow boiling to cease and the sample to cool.

12.1.5. Add 5 mL DI water to the flask to prevent drying during distillation, continue to distill until 50 mL volume is captured.

12.1.6. Rinse the condenser thoroughly with DI water between samples.

12.2. Analysis

12.2.1. Insert samples and reagents into Konelab according to the Konelab SOP (S-MN-I-507).

12.2.2. Select Phenols test PHEN 420.4 and analyze. See Attachment II.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	50 mL aliquot of DI water following the same distillation and analytical steps as the batch samples.	Once per batch of up to 20 samples	Absolute value of the recovery must be less than the reporting limit. Per QAPP or client specifications, alternate criteria such as evaluating to ½ RL may apply.	If outside the acceptance criteria, re-analyze. If reanalysis still does not pass, terminate analysis and reject all batch data associated with the failing MB. The system may need to be recalibrated and the batch re-distilled and re-analyzed. ***Exception:*** If all the affected samples are non-detect or at least 10x the concentration of the failing result, the sample data may be accepted.
Laboratory Control Sample (LCS)	ICV/CCV/LCS Standard following the same distillation and analytical steps as the batch samples.	Once per batch of up to 20 samples	90-110% of the true value	After the instrument has been calibrated, the accuracy of the initial calibration shall be verified and documented by analysis of the LCS solution. When measurements exceed the control limits the analysis must be terminated, the problem corrected and the calibration re-verified.
Matrix Spike & Matrix Spike Duplicate (MS/MSD)	49.975 mL client-provided sample : 0.025 mL QC stock standard = 0.50 mg phenol/L. Follow the same distillation and analytical steps as the batch samples.	One MS/MSD pair per 10 samples with a minimum of one pair per batch	90-110% of the true value RPD $\leq$ 20%	If the spike recovery is not within control limits, and the spike added is greater than or equal to one-forth of the sample result, all data in the project is flagged. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.

14. Data Analysis and Calculations

14.1. See the most current Quality Manual for calculations

14.2. The true value of the Spike Added (SA) is calculated as in the following equation:

$$SA = \frac{(0.025\text{mL})(1000\text{mg/L})}{50\text{mL}}$$

SA = 0.500 mg/L

14.3. Spike sample recovery is calculated as follows and must be within 90–110% recovery:

$$\% \text{ Recovery} = \frac{(SSR - SR)}{SA}$$

Where:
SSR = Spike sample result, mg/L
SR = Sample result, mg/L
SA = Spike added, mg/L

If a sample is negative, the sample should be diluted to remove interferences. If the sample has an MS/MSD associated with it, and the spike did not recover, then the MS/MSD should be diluted and post spiked.

14.4. The relative percent difference (RPD) must be within 20% and is calculated as:

$$RPD = \frac{|S - D|}{\left(\frac{S + D}{2}\right)} x100$$

Where:
S = Sample value, mg/L
D = Duplicate sample value, mg/L

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 & 13.

16. Corrective Actions for Out-of-Control Data

16.1. See tables in section 11 & 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the tables in section 11 & 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study:** An MDL study must be conducted semi-annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Demonstration of Capability (DOC):** Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office**.**

19. Method Modifications

19.1. Not applicable to this SOP

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable to this SOP

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983, Method 420.2.

25.5. U.S. Environmental Protection Agency, "Methods for the Determination of Inorganic Substances in Environmental Samples", August 1993, Method 420.4 revision 1.0

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Phenol Distillation Set-up

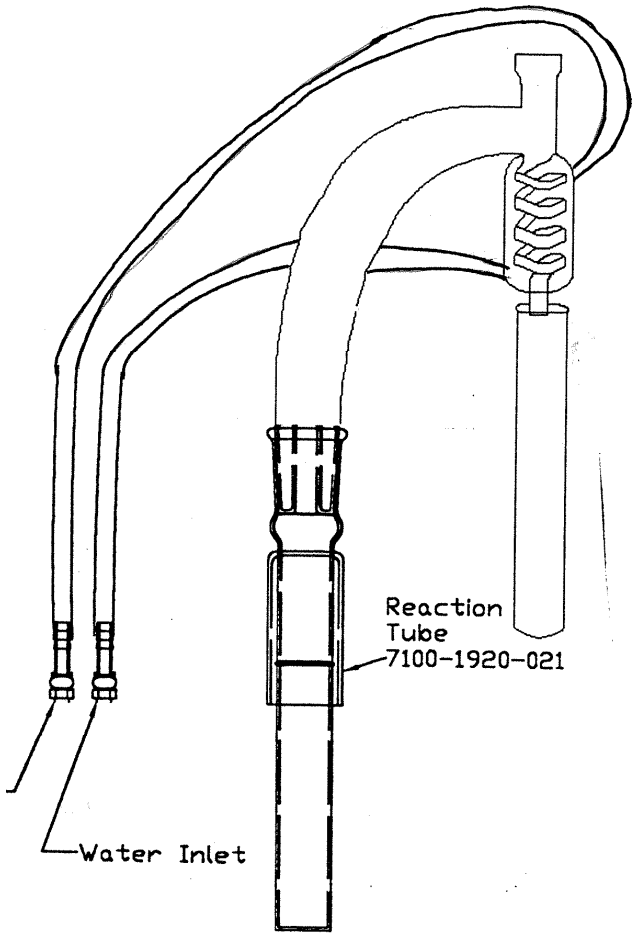
26.2. Attachment II: Test Definition

26.3. Attachment III: Phenol Prep Bench Sheet (example)

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-494-rev.12	Implemented new corp SOP format Attachment III - updated	08Aug2013
S-MN-I-494-rev.13	6.2-6.3 – updated; 6.4 - removed Table 10.1/10.2/13.1 - updated 10.2.1-10.2.6 – updated 10.2.7– added 11.2.3-11.3.3 – deleted 18.2 – edited to semi-annually Removed DoD reference from section 25 Attachment II - updated	14Apr2014

ATTACHMENT I - Phenol Distillation Setup



Ammonia/Phenol/Fluoride
Style

ATTACHMENT II - Test Definition

```
================================================================================
Test definition              AquaKem 6.5                          Page:    1

PHEN 420.4                   Pace Analytical Services, Inc.  MN
                             Analyst:  KEO/AS1/MWD/AAD
                             Instrument: 10WET3
Date :   2014-04-07
Time :   13.15
--------------------------------------------------------------------------------

Full name          Total Phenols EPA 420.4
Online Name                              Test In Use          YES
Test type          Photometric                                LOW     HIGH
                                         Test limit           *       *       mg/l
Result unit        mg/l                  Initial absorbance   *       *       A
Number of Decim.   3                     Dilution limit       *       1.0000  mg/l
                                         Secondary dil 1+     0.0     1.0
                                         Critical limit       *       *       mg/l
                                         Reflex test limit    *       *       mg/l
                                         Reflex test

Acceptance                 Manual        Reference class      LOW     HIGH    In Use
Dilution 1+                0.0

Sample type                Water         Correction factor    1.00
                           Raw water     Correction bias      0.00    mg/l
                           Sewage

Calibration type           Linear
Repeat time (d)            90            Abs error (mA)               *
Points/cal.                Single        Rel error (%)                *
Acceptance                 Manual
Response limit (mA)        MIN           MAX
                           *             *

Bias correction in use     NO

Cd reduction               NO

Type of Calibrators        Series
Calibrator                 Conc.         Dil. ratio
PHE-0                      0.000         1+0.0
PHE-6                      1.000         1+19.0
PHE-6                      1.000         1+9.0
PHE-6                      1.000         1+4.0
PHE-6                      1.000         1+3.0
PHE-6                      1.000         1+1.0
PHE-6                      1.000         1+0.0

Manual QC in Use           YES           Routine QC in Use          YES
Acceptance                 Manual        Interval     Requests      10
                                         Additional condition       NO

Control      Mean          SD            Control      Mean          SD
420.4-CCB    0.00          0.05          420.4-CCB    0.00          0.05
420.4-CCV    0.50          0.05          420.4-CCV    0.50          0.05

Rules in Use               1:1.0*SD      Rules in Use               1:1.0*SD


Blank                      YES
                                         Normal cuvette

Sample                                   Volume (ul)                100
Disp. with                 Extra         Add. Volume (ul)           10
Dilution with              Water         Wash reagent               Water
```

ATTACHMENT II – Test Definition (continued)

```
==============================================================================
Test definition              AquaKem 6.5                          Page:    2

PHEN 420.4                   Pace Analytical Services, Inc.  MN
                             Analyst:  KEO/AS1/MWD/AAD
                             Instrument: 10WET3
Date :   2014-04-07
Time :   13.15
------------------------------------------------------------------------------

Reagent                  420.4 4-AM   Volume (ul)              2
Disp. with               Extra        Add. Volume (ul)         10
Wash reagent             None

Measurement              End point    Blank
Resp. Min(A)             *            Resp. Max(A)             *

Incubation                            Time (sec)               60

Reagent                  420.4 POT    Volume (ul)              4
Disp. with               Extra        Add. Volume (ul)         10
Wash reagent             None

Incubation                            Time (sec)               120

Measurement              End point
Wavelength (nm)          520 nm       Side wavel. (nm)         None
Meas. type               Fixed timing
```

ATTACHMENT II – Test Definition (continued)

Calibration results AquaKem 6.5 Page: 1

Pace Analytical Services, Inc. MN
Analyst: AMO/NMH
Instrument: 10WET3

13.11.2009 14:35

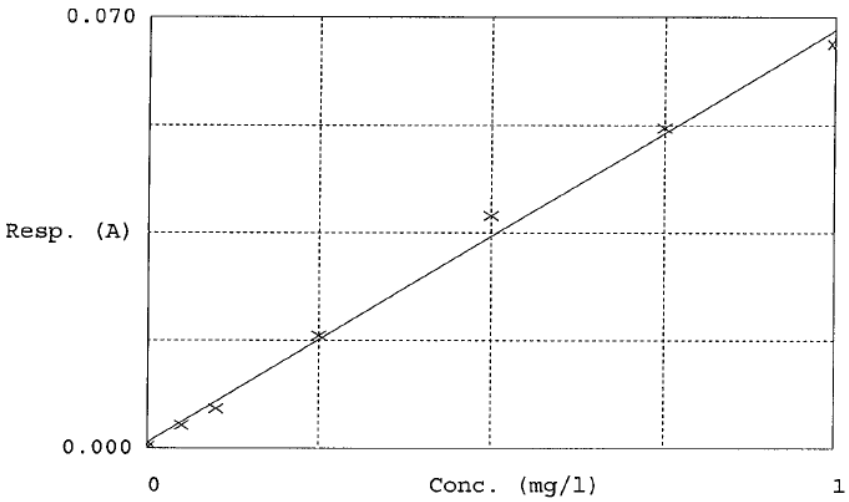
Test PHEN 420.4

Accepted 13.11.2009 14:35

Factor 14.94
Bias 0.001

Coeff. of det. 0.995092

Errors



	Calibrator	Response	Calc. con.	Conc.	Errors
1	PHE-0	0.000	-0.0095	0.0000	
2	PHE-1	0.004	0.0411	0.0500	
3	PHE-2	0.006	0.0816	0.1000	
4	PHE-3	0.018	0.2589	0.2500	
5	PHE-4	0.038	0.5494	0.5000	
6	PHE-5	0.052	0.7628	0.7500	
7	PHE-6	0.066	0.9658	1.0000	
8	420.4-CCB(contr	-0.000	-0.0148	0.0000	
9	420.4-CCV(contr	0.034	0.4960	0.5000	

ATTACHMENT III - Phenolics Prep Sheet (example)

Pace Analytical	Document Name: Phenolics Preparation Sheet	Document Revised: 16May2012 Page 1 of 1
	Document Number: F-MN-I-135-rev.06	Issuing Authority: Pace Minnesota Quality Office

Method:	Click to select	Queue:	WETA
Instrument ID:	10WET3	Batch:	
Analyst:		Date:	
Reviewed By:		Reviewed Date:	

Sample	Sample Vol. (mL)	Collected Vol. (mL)	pH Adjusted to 4? Y or N	Comments
	50	50		
	50	50		
	50	50		
	50	50		
	50	50		
	50	50		
	50	50		
	50	50		
	50	50		
	50	50		
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	50	50		
	50	50		
	50	50		

All samples and QC are distilled to a final volume of 50 mL.

Reagent	Vendor	Lot/Log Number
QC 1,000 ppm Std	Ricca	
1N NaOH Solution	Pace	

Distillation Temp:	190°C
Block(s) Used:	10WET: ☐ 12 ☐ 13 ☐ 14
Pipet ID:	
Syringe ID:	

Pace Analytical Services, Inc.
1700 Elm Street SE, Suite 200
Minneapolis, MN 55414

Phone: 612-607-1700
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STANDARD OPERATING PROCEDURE

DETERMINATION OF HEXANE EXTRACTABLE MATERIAL (HEM) AND SILICA GEL TREATED-HEXANE EXTRACTABLE MATERIAL (SGT-HEM) IN WATER

Reference Methods: EPA 1664A

Local SOP Number:	S-MN-I-520-Rev.13
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-520-Rev.12

APPROVALS

Laboratory Assistant General Manager — Date: 7/18/2014

Laboratory Quality Manager — Date: 18 Jul 2014

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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1. Purpose/Identification of Method

1.1. This Standard Operating Procedure (SOP) describes the gravimetric determination of n-hexane extractable material (HEM; oil and grease) and silica gel-treated n-hexane extractable material (SGT-HEM; total petroleum hydrocarbon) in surface and saline waters and industrial and domestic aqueous wastes by solid-phase extraction and liquid/liquid extraction followed by gravimetric measurement. This SOP follows EPA Method 1664A.

2. Summary of Method

2.1. Solid-phase extraction (SPE): A nominal 1-liter aliquot of aqueous sample is acidified to pH 2.0 with 1:1 HCl solution and filtered through a solid phase extraction disk. The disk is then serially extracted three times with n-hexane. The extract is filtered through a phase separator filter paper and evaporated. The HEM extract residue is then desiccated and weighed.

2.2. Liquid/liquid extraction (LLE; Separatory funnel): A nominal 1-liter aliquot of aqueous sample is acidified to pH 2.0 with 1:1 HCl solution and is serially extracted with hexane in a separatory funnel. The solvent is evaporated from the extract and the residue weighed.

2.3. For SGT-HEM determination the residue is re-dissolved in hexane and an amount of silica gel is added to the solution to remove polar materials. The solution is filtered, evaporated, desiccated, and weighed.

3. Scope and Application

3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method.

3.2. Parameters: Extractable materials that may be determined are relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and related materials. The term “n-hexane extractable material” reflects that this method can be used to determine materials other than oils and greases such as sulfur compounds, organic dyes and chlorophyll.

3.3. This procedure is not applicable to measurement of materials that volatilize at temperatures below 85ºC. Petroleum fuels from gasoline through #2 fuel oil may be partially lost during the solvent removal process. In addition, some crude oils and heavy fuel oils may contain materials that are not soluble in n-hexane, thus recoveries of these materials may be low.

4. Applicable Matrices

4.1. This SOP is applicable to surface and saline waters and industrial and domestic aqueous wastes.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes is 5 mg/L for this method. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Interferences extracted from samples will vary considerably from source to source, depending upon the nature of the site being sampled. A smaller sample volume may be needed for extraction for samples which consist of complex matrices containing substances such as particulates or detergents that may interfere with the extraction procedure.

6.2. This SOP is applicable to samples containing HEM in concentrations from 5-1000 mg/L. If clients consistently send samples with residue that exceeds the upper detection limits of 1000 mg/L, reduced volumes may be requested through the customer service/sales department, allow for results within the detection range of 5 to 1000 mg/L of residue.

6.3. Hexane has the ability to dissolve not only oil and grease but also other organic substances. No known solvent will dissolve selectively only oil and grease. Solvent removal results in the loss of short-chain hydrocarbons and simple aromatics by volatilization.

6.4. The filter paper may have the potential to inflate results and must be rinsed with hexane prior to use.

6.5. Silica gel has the potential to inflate results for SGT-HEM if it is transferred with the hexane into the pan for gravimetric determination.

6.6. All solvents must have an evaporated residue of < 1.0 mg HEM/L.

6.7. Samples with high sulfur content may interfere with results due to the solubility of sulfur in hexane.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Collection, Preservation, Storage and Holding Time Table

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Samples are collected and stored in amber glass bottles with Teflon-lined caps following conventional sampling practices, except that the bottle must not be pre-rinsed with sample before collection.	Samples must be preserved with HCl or H_2SO_4 to a pH of less than 2.0	Above freezing but below 6°C	28 days of collection

7.2. The high probability that extractable matter may adhere to sampling equipment and result in measurements that are biased low precludes the collection of composite samples for determination of oil and grease. Therefore, samples must be collected as grab samples. If a composite measurement is required, individual grab samples collected at prescribed time intervals must be analyzed separately and the concentrations averaged.

7.3. Since this test consumes the entire contents of the sample bottle it is highly recommended that each sample is collected in duplicate.

7.4. By default the method prescribes the collection of 1 liter of sample. However, if a sample is known or suspected to contain greater than 500 mg/L of HEM, a proportionally smaller volume of sample may be collected in a glass bottle. The volume required will depend upon the estimated amount of HEM.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Equipment and Supplies Table

Supply	Description	Vendor/ Item # / Description
Disposable funnel with extraction disk	47 mm extraction disk	Environmental Express # G5247, or equivalent
Aluminum dishes	70 mL smooth-sided or 200 mL fluted	Fisher # 08-732-103 or 08-732-108, or equivalent
Stainless steel funnel	Used to remove water or solids from extract	Fisher Scientific or equivalent
Phase separator filter paper	Used to remove water or solids from extract	Fisher Scientific # 2200 125, or equivalent
Separatory funnels	2-L, with Teflon stopcocks	Fisher Scientific or equivalent
Glass beakers	Various sizes, used in liquid/liquid extraction to remove emulsion from extract	Fisher Scientific or equivalent
Solid Phase Extraction Manifold	Expanded Extraction Station	J.T. Baker, or equivalent
Solvent Evaporation Unit	Speed VAP II	Horizon Technology, or equivalent

Pipettes	Glass, 5-mL. Used to add acid to sample, if necessary	Fisher Scientific # 1367835A, or equivalent
pH Strips	0-14 pH range. Manufactured by Whatman	Fisher Scientific # 987617
Glass graduated cylinders	Class A. 1000 mL or 500 mL, used for sample volume determination	Fisher Scientific, or equivalent
Analytical balance	Capable of weighing 0.1 mg	Fisher Scientific or equivalent
Stir plate and magnetic stir bars	Used in SGT-HEM extraction	Fisher Scientific, or equivalent
Desiccator with Drierite	Used in final drying process	Fisher Scientific or equivalent
Microsoft Excel	Spreadsheet software	See master list for current version
EPIC Horizon	Data Reporting Software (LIMS)	See master list for current version

10. Reagents and Standards

10.1. Target analyte solutions, surrogate solutions, and matrix spike solutions are obtained from various vendors and verified for accuracy. Internal standard solutions are also obtained from vendors in solution form. These solutions are stored per manufacturer's specifications, and have an expiration date of one year after being opened or the manufacturer's expiration date if that date is prior to the one year date.

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
De-ionized (DI) Water		Verify daily that pH and specific conductance are within acceptable limits.
Hexadecane/Stearic Acid Standard	Snip and pour standard. 40 mg HEM, 20 mg SGT-HEM	Environmental Express part # G3025
Second Source Hexadecane/Stearic Acid Standard	EPA Method 1664 LCS Solution, 40 mg/L	ChromaneX, 9220-001, or equivalent replacement
n-Hexane	< 1 mg HEM/L residue. Store at room temperature. Expires one year after opening.	Fisher part # H303-4 or equivalent
Acetone	< 1 mg HEM/L residue. Store at room temperature. Expires one year after opening.	Fisher part # A929-4 or equivalent
Methanol	< 1 mg HEM/L residue. Store at room temperature. Expires one year after opening.	Fisher part # A454-4 or equivalent
Hydrochloric Acid (HCl)	6.00N. Store at room temperature. Expires one year after opening.	Fisher part # 3750-1 or equivalent
Sulfuric Acid (H_2SO_4)	Concentrated; dilute 1:3 to create ~6N concentration prior to adding to a sample. Store at room temperature. Expires one year after opening.	Fisher part # A300SI-212 or equivalent
Red food coloring	Used to color water for easy removal from extract. Store at room temperature. Does not expire.	Purchase from grocery store
Sodium Sulfate (Na_2SO_4)	Anhydrous granular, 10-60 mesh. Bake for 4 hours at 400 ^oC. Store in a tightly closed glass container at room temperature.	Fisher part # S415-200LB or equivalent
Sodium Chloride (NaCl)	Used to remove emulsion during LLE extraction.	Fisher part # S640-500 or equivalent
Silica Gel	Anhydrous, 50-150 µm mesh. Dry at 200-250 ^oC for a minimum of 24 hours and store in a desiccator or tightly sealed container. NOTE: 75-150 mesh is not available.	Fisher part # 04669-1 or equivalent
Drierite	Desiccant. Store at room temperature in a tightly closed glass jar. Drierite is no longer effective when blue crystals turn pink. To regenerate used Drierite,	Fisher part # 075773B Blue indicator: Fisher part # 075783B

	see SOP S-MN-O-557, Drierite Regeneration (or equivalent replacement).	

10.1.1. Sodium Sulfate: Bake for 4 hours at 400ºC. Store in a tightly closed glass container at room temperature.

10.1.2. Silica Gel: Bake for at least 24 hours at 200-250ºC. Store in a tightly closed glass container at room temperature.

10.1.3. Drierite: Use a mix of white and blue crystals in a closed desiccator. When the blue crystals begin to turn purple or pink, the drierite is losing its effectiveness and must be replaced or regenerated. See S-MN-O-557 (or equivalent replacement) for drierite regeneration procedure.

11. Calibration and Standardization

11.1. Daily calibration of the balance is required following SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Record in associated balance calibration logbook. Calibration limits are found in the balance calibration logbook; if values exceed these limits, recalibrate the balance.

11.2. All balances must be certified by an outside agency on an annual basis with documentation of the calibration maintained in the QA office.

11.3. Per method EPA 1664 revision B, section 9.5, calibration of the balance must be verified before and after each analytical batch.

12. Procedure

12.1. Determination of Procedure

12.1.1. Evaluate each sample to determine whether it can be extracted for HEM by SPE or by separatory funnel. Due to speed and ease, SPE is preferred; however, the following characteristics of a sample matrix may necessitate extraction by separatory funnel:

12.1.1.1. Very thick, cloudy or chunky matrices.

12.1.1.2. Heavily sedimented matrices. Note: Depending on the amount of sediment present, it can be allowed to settle, the water decanted into the SPE funnel and the sediment roused and added towards the end of extraction.

12.1.1.3. Matrices in which oil or fats are visibly present.

12.1.1.4. Reactive matrices. Note: These are generally clear in appearance but tend to have an alkaline pH and will begin foaming during pH adjustment (12.2.2).

12.1.1.5. Matrices containing surfactants, detergents or other chemicals. Note: These are generally clear in appearance but tend to have an alkaline pH. They are sometimes reactive as noted in 12.1.1.4.

12.2. Sample & QC Preparation

12.2.1. Allow samples to warm to room temperature.

12.2.2. Verify the pH of the sample is less than 2 using the following procedure:

12.2.2.1. Using a glass stir rod, allow one drop of the sample to drip on the pH paper.

12.2.2.2. If the pH is 2 or less, proceed to 12.2.3. If the pH is greater than 2, add 6N HCl or 6N H2SO4 dropwise to the sample. Between each aliquot of acid added, place the cover on the container and invert several times to mix. Remove the cover and repeat step 12.2.2.1. Repeat this process until the pH is less than or equal to 2. Rinse the rod with hexane into the extractor after the pH check is complete.

12.2.2.3. Note: DO NOT over-acidify the samples. This will cause the disk material to breakdown and may result in low HEM recoveries.

12.2.3. Mark the line of meniscus on the outside of the sample bottle. The sample volume will be measured by filling the empty container with tap water to the mark and subsequently pouring the water into a Class A graduated cylinder. This is done after extraction when using the SPE or during extraction when using separatory funnels. Record the volume in the electronic prep log (F-MN-I-321).

12.2.4. Prepare the QC samples:

12.2.4.1. Fill a one liter bottle half way with DI water then add 3 mL of 6N HCl or 6N H2SO4. Fill to top with DI water. This is used as the Method Blank (MB).

12.2.4.2. Fill a one liter bottle half way with DI water then add 3 mL of 6N HCl or 6N H2SO4. Add the contents of one tube of hexadecane/stearic acid standard to the bottle. Rinse tube with acetone and pour into container. Mix gently and fill to top with DI water. This is the Laboratory Control Sample (LCS).

12.2.4.3. For the MS/MSD, add the contents of one tube of hexadecane/stearic acid standard to designated sample containers.

12.2.4.4. If there is not enough headspace available to spike into the sample container, add the contents of the hexadecane/stearic acid tube to the sample after it has been poured into the separatory funnel for liquid/liquid extraction. For SPE, add the tube contents after filtering approximately 10-20 mL of straight sample.

12.2.5. Aluminum Dish Preparation

12.2.5.1. Label one aluminum dish for each sample and QC sample.

12.2.5.2. Using the analytical balance, obtain a tare weight for each dish and record it in the electronic prep log (F-MN-I-321).

12.3. Determination of HEM by SPE

12.3.1. Place Speedisk on manifold and close all valves.

12.3.2. Add 10-15 mL (above ridgeline) of acetone, open valve a slight turn then close. Allow soaking for ~2 minutes, vacuum dry.

12.3.3. Add 10-15 mL hexane, open valve a slight turn then close. Allow soaking for ~2 minutes, vacuum dry.

12.3.4. Add 10-15 mL methanol, open valve a slight turn then close. Allow soaking for ~2 minutes. DO NOT ALLOW TO GO DRY!

12.3.5. Add sample to the funnel by inverting bottle and placing onto funnel or filling the funnel to the top. If sample contains solids, decant onto disk then filter solids when sample is almost completely filtered.

12.3.6. Filter the entire sample through the disk NEVER allowing the disk to go dry during the extraction process.

12.3.7. Continue to vacuum filter after sample is done to dry, ~3-5 minutes.

12.3.8. Place a 40 mL vial in the collection chamber and place onto the manifold with the extraction filter on top.

12.3.9. Rinse sample bottle with ~10 mL of hexane and pour onto disk, open valve slightly then close. Allow soaking for ~2 minutes then open valve and collect hexane in vial. Repeat two more times.

12.3.10. Filter the vial containing the extract through hexane-rinsed phase separator filter paper collecting it in a pre-weighed aluminum dish that is labeled with the sample ID. Rinse the vial with ~5 mL hexane and add to the aluminum dish. Repeat the rinse process once more.

12.3.11. Place the dish on a hot plate set at 40ºC in a fume hood to evaporate solvent from pan. When evaporation is complete, transfer the dishes to a desiccator for at least 30 minutes to allow them to come to room temperature. Proceed to section 12.5.

12.4. Determination of HEM by Separatory Funnel

12.4.1. Pour the sample into a 2-liter separatory funnel, making sure the stopcock is in the closed position.

12.4.2. Rinse the sample bottle with a 30-mL portion of hexane, pouring the washings into the separatory funnel. Cap the funnel and vent at least twice into the fume hood. Manually shake the separatory funnel for 2 minutes.

12.4.3. The separatory funnel may be tumbled for 3 minutes for the last two extractions.

12.4.4. Allow the layers to separate in funnel. If an emulsion forms between the phases, and the emulsion is greater than one-third the volume of the solvent layer, add a few grams of sodium chloride to the separatory funnel.

12.4.5. Allow the sample to stand and separate. Loosen the lid of the separatory funnel to allow venting and open the stopcock to drain the aqueous layer back into the sample bottle. Drain as much of the aqueous layer as possible without losing any of the hexane layer.

12.4.6. Decant hexane layer into a 500 mL (or greater) glass beaker.

12.4.6.1. If the sample contains solid material or any of the aqueous layer, it will be necessary to add Na2SO4. Using a clean tongue depressor, mix small amounts of Na_2SO_4 into the beaker until the aqueous layer and/or solid material have been absorbed.

12.4.6.2. Decant the hexane layer into a pre-weighed aluminum dish labeled with the sample ID. If the hexane layer contains significant amounts of solids, pour it through a funnel and phase separator filter paper that have been pre-rinsed with hexane. Rinse the beaker with an additional 10-20 mL of hexane and drain through the funnel into the dish.

12.4.6.3. Red food dye may be used to dye any water that may be present to aid in the removal of the aqueous layer during extraction. Take care to remove only the aqueous layer.

12.4.7. Repeat steps 12.4.1 through 12.4.5 two more times.

12.4.8. Place the dishes on a hot plate set at 40ºC in a fume hood to evaporate solvent. When evaporation is complete, transfer the dishes to a desiccator for at least 30 minutes to allow them to come to room temperature. Proceed to section 12.5.

12.5. Gravimetric Determination of Results

12.5.1. Weigh each dish to 0.1 mg and record the weight in the electronic prep log (F-MN-I-321).

12.5.2. Weigh each dish again and record. If the second weight is within ± 0.5 mg of the first, constant weight is established and analysis is complete, otherwise repeat this step for a third weight and, if necessary, a fourth weight.

12.5.2.1. If any weight measurement agrees within 4% of a previous weight, it is considered a constant weight and analysis is complete.

12.5.2.2. If the fourth weight is still not within ± 0.5 mg, use the fourth weight and qualify the sample data as not having established constant weight.

12.6. Determination of SGT-HEM

12.6.1. Follow the procedure as outlined in section 12.3 or 12.4.

12.6.2. Thoroughly rinse the dish used for HEM with hexane to collect all residue present in the dish and transfer to a large fluted aluminum dish.

12.6.3. Add ~3 g silica gel for every 100 mg of HEM residue in the large fluted aluminum dish. Add a stir bar.

12.6.4. Stir for ~5 minutes then sit for ~1 minute to allow silica gel to settle. Pour through hexane-rinsed phase separator filter paper collecting it in the same aluminum dish used for HEM determination.

12.6.5. Place the dish on a hot plate set at 40ºC in a fume hood to evaporate the solvent. When evaporation is complete, transfer the dish to a desiccator for at least 30 minutes to allow it to come to room temperature.

12.6.6. Weigh to a constant weight as in section 12.5.

13. Quality Control

13.1.QC Table 13.1

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	DI water	Once per batch of up to 20 samples.	Must contain less than 5 mg/L HEM or SGT-HEM.	If the method blank does not meet this criterion, the source of the contamination must be investigated and documented, and attempts made to eliminate the source. Reanalyze all samples associated with the failed method blank. If sufficient sample volume is not available to reanalyze samples, report the sample data with a qualifier on each sample associated with the non-compliant method blank. ***Exceptions:*** If sample results are non-detect, report results without qualification. If sample results are 10x or greater than the MB result, report results and narrate on the validation report as not impacted by the blank contamination. If sample results are less than 10x the MB result and the sample cannot be reanalyzed, report the results with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	DI water with one tube of hexadecane / stearic acid standard added.	Once per batch of up to 20 samples. If there is insufficient sample volume for matrix spike and duplicate analysis, an LCSD must be performed.	HEM recovery must be within 78-114% of the 40 mg/L true value. SGT-HEM recovery must be within 64-132% of the 20 mg/L true value. RPD requirements of the LCS and LCSD: HEM $\leq$ 18% SGT-HEM $\leq$ 34%.	If the recovery is not within control limits, check for errors in calculations, standards preparation and spiking of the LCS solution. If an error or problem is found and can be corrected by amending the calculations and the result falls within the limits, accept the data and report without a qualifier flag. If no errors are found, re-prep and re-analyze the LCS as long as re-analysis occurs on the same day as the original analysis. If re-analyzed LCS fails, re-analyze the all samples associated with the failed LCS. If sufficient sample volume is not available to re-analyze samples, report the sample data with a qualifier on each sample associated with the non-compliant LCS. ***Exceptions:*** If the LCS recovery is greater than the acceptance criteria and the sample results are non-detect, the sample data may be reported with appropriate data qualifiers.

Second Source Quality Control Sample	DI water with second source hexadecane/stearic acid standard added	1 per month	HEM recovery must be within 78-114%. SGT-HEM recovery must be within 64-132% .	If the recovery is not within control limits, check for errors in calculations, standards preparation and spiking of the solution. Review the LCS for similar bias. If the LCS passes and the second source fails, one or both solutions are in question. Obtain new standards for verification, prior to proceeding with analysis. Determine the source of the bias.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	Client-provided sample with one tube of hexadecane / stearic acid standard added.	Once per batch if sufficient volume of sample is available. See 19.3. An MSD can be performed per client request and may take the place of a sample duplicate (DUP).	HEM recovery must be within 78-114% of the 40 mg/L true value. SGT-HEM recovery must be within 64-132% of the 20 mg/L true value. RPD requirements of the MS and MSD: HEM $\leq$ 18% SGT-HEM $\leq$ 34%.	If the recovery is not within control limits or the RPD exceeds control limits in the case of an MS/MSD, check for errors in calculations, standards preparation and spiking of the MS solution. If an error or problem is found and can be corrected by amending the calculations and the result falls within the limits, accept the data and report without a qualifier flag. If no errors are found, report the sample data with a qualifier on the MS and parent sample. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.
Duplicate Sample (DUP)	Client-provided sample.	Once per batch if sufficient volume of sample is available. An MSD may be performed in place of a DUP.	RPD requirements of the parent sample and DUP: HEM $\leq$ 18% SGT-HEM $\leq$ 34%.	If the RPD exceeds control limits, check for errors in calculations and sample preparation. If an error or problem is found and it can be corrected by amending the calculations and the result falls within the limits, accept the data and report without a flag. If no errors are found, report the sample data with a qualifier on the DUP and parent sample.

14. Data Analysis and Calculations

14.1. HEM or SGT-HEM Recovery

$$\text{HEM or SGT - HEM, mg/L} = \frac{(\text{F - I})}{V} \times \frac{1000mL}{L}$$

Where:
F = Final dish weight, mg
I = Initial dish weight, mg
V = Volume of sample, mL

14.2.LCS and MS/MSD % Recovery

$$\%\,\text{Recovery} = \frac{SS - PS}{SA} \times 100$$

Where:
SS = Spiked Sample result, mg/L
PS = Parent Sample result, mg/L (only applicable to MS/MSD, LCS = 0)
SA = Spike Added from spiking standard, mg/L

14.3. Relative Percent Difference (RPD)

$$RPD = \frac{|PS - Dup|}{(PS + Dup)/2} \times 100$$

Where:

PS = Parent Sample result
Dup = Duplicate sample result

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See table in section 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See table in section 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 (or equivalent replacement), Method Detection Limit Studies for each matrix per instrument

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) S-ALL-Q-020 (or equivalent replacement), Training Procedures.

18.4. **Periodic performance evaluation (PE)** samples are analyzed periodically to demonstrate continuing competence per SOP S-MN-Q-258 (or equivalent replacement). Results are stored in the Quality office.

19. Method Modifications

19.1. Red food coloring may be added to sample extract prior to drying. The purpose of the food coloring is to stain the water-soluble (non-HEM) layer a deep red, thereby allowing the analyst a greater ease of distinguishing the aqueous layer from the organic layer.

19.2. Evaporation of the solvent at 40ºC on a hot plate has been demonstrated to be an effective method modification according to SOP S-MN-Q-269. See sections 12.2.11, 12.3.7 and 12.5.5.

19.3. According to EPA 1664 W Revision B, Section 9.3, “The laboratory must spike a minimum of 5 percent of all samples from a given sampling site or, if for compliance monitoring, from a given discharge/waste stream (matrix type).” The laboratory is unable to control the amount of volume submitted for this test and therefore cannot strictly follow the 5% spiking rule due to insufficient sample volume for spiking and/or the inability to re-analyze a sample in the event of QC failure.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Refer to documentation provided with instrument for troubleshooting techniques.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

22.3. n-Hexane has been shown to have increased neurotoxic effects over other hexanes and some other solvents. Inhalation of n-hexane should be minimized by performing all operations with n-hexane in a hood or well-ventilated area.

22.4. n-Hexane is extremely flammable and must be kept away from sources of heat and open flame.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003, Waste Handling, or equivalent replacement.

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. EPA Method 1664, Revision A, "N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry)" EPA-821-R-98-002, February, 1999

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-520-Rev.11	Removed DoD reference from section 25 Added MN Admin contract in 13.1 Reworded MB 'exception' comment in 13.1 10.1—updated description and vendor; added storage criteria Added 10.1.1 – 10.1.3 (Sodium sulfate, silica gel and drierirte) 11.1—modified to create 11.2 11.3—added 12.1.2.2—added "6N" 12.1.4.1, 12.1.4.2—added "6N H2SO4" 12.1.4.2, 12.1.4.2—added "hexadecane.." 13.1—criteria and frequency updated 13.1—MS criteria updated 19.1—modified 19.3—added and updated Attachment I - updated	06Feb2014
S-MN-I-520-Rev.12	12.1.4.3—added "containers" 12.1.4.4—added "If there is…of straight sample."	19Mar2014
S-MN-I-520-Rev.13	10.1 Added Chromanex standards 12.1 – Determination of Procedure added 12.6.2 – Revised 12.6.3 – added "in the large…" 12.2.5.2/12.5.1 – changed to record it in the electronic prep log (F-MN-I-346) instead of Attachment I. 12.3.11/12.4.8 – added for at least 30 mins… 12.5.2 – minor content edited 12.6.5 – removed 12.3 from text Section 13 added the use of a second source quality control on a monthly basis 26.1 – attachment removed	17Jun2014

APPENDIX H

Resumes for Severson

Resumes for Robert E. Lee

Years of Experience:
18

Education:

- BA – Marketing, The University of Alabama, 1992
- MBA, Management, The University of Tennessee at Chattanooga, 1994

Certifications:

- OSHA 40 Hour HAZWOPER Training
- OSHA 8 Hour HAZWOPER Refresher
- USACE – Construction Quality Management

Role and Responsibility:

- Responsible for delivery of completed project in compliance with plans and specifications.
- Attends all scheduled progress meetings.
- Meets daily with the project team to review planned activities.
- Develops and maintains all project deliverables, submittals, schedule and reports.
- Develops and manages cost control and accounting, procurement and expediting material delivery, inspection, and quality control functions.

Experience and Expertise:

- Remediation skills developed over 18 years of remedial construction project experience.
- Understands CERCLA, RCRA, TSCA and additional regulatory requirements.
- Firm Fixed Price, Cost Reimbursable, Fixed Fee, Guaranteed Maximum Price Contract Experience
- Union and Open Shop Labor
- Excavation and Earthwork
- Steel Sheeting Systems
- Recovery and Treatment Systems
- Solidification/Stabilization

Jeffrey Scott Burns

Project Controls Manager

Past Project Experience

DA Collins/Dredge Mohawk River/Scotia, NY: Project Accountant for a NYS DOT reclamation project as a result of damages to the Erie Canal during Hurricane Irene. The Manual for Uniform Recordkeeping (MURK) was used for daily reporting. All incurred cost was documented and reported on the prescribed forms (MURK). Daily meetings with the Prime Contractor and state project manager to ensure that all costs and forecast were timely and accurate.

Spring Valley Site, (Phases I, II and III), Washington, DC: Project Manager/Accountant for the removal of arsenic contaminated soil on 69 residential properties under USACE Time Critical Removal Action regulations. Contaminated soil was excavated using surgical techniques. Complete restoration of the disturbed sites included backfill and topsoil placement; sod placement and care; replacement of driveways and sidewalks; and installation of additional landscape features such as retaining walls and plantings. Preparation of weekly costs, to include daily, weekly, PCSR, WVN's projections and close out reports. Also responsible for all procurements. Sevenson served as the coordinator between the USACE, residential homeowners, and subcontractors.

Montclair/Glen Ridge/West Orange Radium Sites, Phases IIB-V, Essex County, NJ: Project Engineer. Project involved the low-level radiological remediation of residential and industrial properties resulting in the excavation and shipment of 194,000 cy of low-level radiological waste materials to the EnviroCare LLRW facility in Utah. Remedial actions ranged from selective to extensive demolition; excavation and removal of contaminated materials; reconstruction and site restoration. Use of sophisticated shoring and underpinning systems to stabilize homes. Successful community relations plan resulted in numerous letters of commendation from federal, state, community leaders as well as home owners.

US Radium Phase I & II, Orange, NJ: Assistant QC Manager for the complete or partial demolition and restoration of 188 commercial, industrial, and residential properties. Excavation, characterization and T+D of 73,000 cubic yards of low level radioactive soils to USACE's designated disposal facility via railcar. Design, construction, and operation of an innovative site dewatering and construction water treatment system rated at 350 gpm. Multi-media sampling and sample management of soils, air, water, and demolition debris. Successful community relations plan focused on an open door policy and resulted in numerous letters of commendation from federal, state, community leaders as well as home owners. Extensive restoration of affected properties.

Lipari Landfill, Pitman, NJ: QA/QC Engineer. Multi-phased project required the installation of 1.5 miles of double-wall HDPE force main, complete with electronic leak detection; over 2 miles of temporary haul roads, including stream crossings; diversion of Chestnut Branch; and construction of soil erosion and sediment control measures. Excavation of 60,000 yd^3 of VOC-contaminated soils from marsh areas using specialty amphibious excavating and hauling equipment. Soils were treated using LTTD then placed in an on-site cell. Wetlands areas were restored to natural conditions with native tidal marsh vegetation; and a drain bordering the marsh was installed to protect off-site areas from re-contamination from the adjacent landfill. Installation of a surface water bypass channel, diversion berms, and floating haul roads on the exposed lake surfaces, followed by excavation of 85,000 yd^3 of contaminated lake sediments. The lake was restored to depths and contours in accordance with USACE guidance specifications.

Years of Experience:
30

Registrations and Certifications:

- Six Sigma Black Belt
- OSHA HAZWOPER 40 Hour Training
- OSHA Supervisors Training
- First Aid/CPR Training
- USDOE Radiation Worker II Certification

Highlights of Qualifications:

- 30 years' experience managing remediation projects including river sediment with PCBs
- Design, engineering, planning, constructability and operations of processing facilities
- Horizontal auger and cutterhead
- Specialized pumping systems, and associated manifolds, valves, controls, and pipeline systems
- Specialized programs to compile and monitor system input, process, and throughput data
- Winops and Hypack systems
- Specialized equipment and methods for in-situ and ex-situ stabilization and solidification operations
- Superfund Sites and RCRA Facilities project management
- Oversight and implementation treatability studies
- Oversight of radioactive waste remediation and MGP sites
- Expertise in mechanical, hydraulic dredging, solids/liquid separation, and large volume WWT
- Member of the Wisconsin DNR & EPA Fox River Technical Review Team (panel of experts)

Michael D. Crystal

Corporate Program Manager

Past Project Experience

Menominee River Sediment Removal Project, Marinette, WI: Program Manager. Mechanical environmental dredging of approximately 235,000 cy of river sediment. Dredged sediments loaded onto hopper barges. Barges transport sediments to an offloading and storage area. Stabilization of sediments using a pugmill. Processed sediment for offsite transportation and disposal. Dredge water treated in WWTP prior to discharge.

Sheboygan Harbor Dredging, Sheboygan, WI: Program Manager. Project conducted by USEPA's Great Lakes National Program Office at the inner harbor of this active community and required detailed community relations plan and health and safety controls. Mechanical dredging of approximately 170,000 cy of PCB-contaminated sediments. Stabilization of sediments using Class C fly ash was required to condition material for offsite transportation and disposal. Perimeter misting system was installed to minimize dust missions. All water generated from dredging, stabilization and decon operations was treated onsite through a 200 gpm WWTS.

Onondaga Lake Dredging, Sediment Transportation and Capping Project, Syracuse, NY: Program Manager. Project requires Sevenson to provide design support of a dredging, sediment, capping, and transport system. Dredge volume estimated at 2.4 million cy of sediments. Capping will extend over approximately 600 acres. Approximately 2,100,000 cy of material will be removed using a hydraulic dredge and a mechanical excavator with a hydraulic clam bucket. Upon completion of dredging operations, Sevenson will perform backfilling and capping operations. Material barges will transport material to the backfilling and capping areas and an excavator with a clamshell bucket will be used to place the materials into a slurry system used to install the cap.

Strontia Springs Reservoir Sediment Removal, Littleton, CO: Program Manager for the hydraulic dredging a target in-situ volume of 625,000 cy of sediments from the upstream portion of Strontia Springs Reservoir; pumping the sediments to a sediment management site at the Kassler Sand Filter Beds; dewatering the slurry; treating and discharging carrier water; placing dewatered sediment in the Sediment Management Area; and seeding and stabilizing the sediment spoils piles.

Ohio River Sediment Removal and Restoration, Follansbee, WV: Program Manager. Project required mechanical dredging of 30,000 CY of sediment contaminated with coal tar utilizing a barge mounted PC800 excavator with a 2 CY Young clam bucket. Dredged sediment was loaded into scows and delivered to the plant dock where it was

transferred to a sediment dewatering pad for stabilization with Portland cement. Water generated from work activities was collected and treated onsite. An existing timber piling dock was demolished. Dredging, demolition, debris removal, and sediment capping was conducted on an active part of the Ohio River. Additional marine equipment included a 1400 HP and 600 HP tugs, five large sediment transport barges, and three large capping barges. Cap material was comprised of filter stone and riprap. A Hypack positioning system was used to place cap material. Sevenson utilized two dive crews working 24 hours a day to install 235,000 SF of CETCO reactive Organoclay mat. Sevenson closely communicated with the plant personnel to move and redirect barges to accommodate for plant delivers and sediment removal activities.

Value–Added Benefits:

- Established EPA Region II and NYSDEC relationships
- Saved client $10 million in capital savings through alternative approach
- Implementation of community communications plan to alleviate concerns regarding contaminated sediment clean-up
- Managed dozens of projects with zero lost time or significantly reduced lost time
- Designed, installed and operated Pilot Test equipment capable of 100% scale up accuracy

TVA Emory River Dredging and Ash Processing, Harriman, TN: Program Manager for the hydraulic dredging and processing of 2,500,000 cy of dredged fly ash and sediment. Installed and operated (5) cutterhead dredges between 14 inch and 20 inch pump sizes. Installed 8 booster pumps capable of pumping high solids slurry's up to 3 miles away. Achieved average production rates of 12,000 cy/day. Operated an ash recovery and processing system using 12 large excavators (PC800, PC600, PC 400), 6 dozers, and twelve large off road haulers. Operations ran 24-hours per day six or 7 days per week. Sevenson instituted an in house survey program for taking daily surveys and sharing the raw data with the TVA construction manager for quick and accurate daily production volumes. Installed and operated a state-of-the-art fully automated RTK GPS system for controlling clean up passes and minimizing over dredging.

New Bedford Harbor Superfund Site, New Bedford, MA: Program/ Site Manager for a $15-20 million project that included the dredging, desanding, dewatering, and wastewater treatment of 900,000 CY contaminated sediment. Responsibilities included direct oversight for sampling, treatability studies, design, equipment selection, and staffing of project. As a marine tidal estuary, project challenges included cathodic protection and corrosion. Specialized equipment and techniques were designed and installed to accommodate the long distance slurry transfer pumps and pipelines anchored to the bottom of the harbor. Extensive re-fabrication and coatings were developed to ensure the longevity of the equipment. Mobilization was completed within 12 weeks of a multiple dredge system containing over 20,000 lf of piping. The wastewater treatment system was designed to handle 2,000 gpm with metals and PCB discharge limits in the ppt range. Best Value Scenario resulted in $10 million in capital savings for client and 1 year earlier start.

Kress Creek/Dupage River Superfund Site, West Chicago, IL: Program/Site Manager for the removal of radioactive thorium contaminated uplands soils and sediments in the Kress Creek and Dupage River. An aggressive waste minimization program was implement through constant gps survey monitoring and selective excavations to minimize the volume of radioactive waste being hauled offsite for disposal. Implemented integrated team approach to reduce redundancy in personnel resulting in significant cost savings.

Fox River SMU 56/57 PCB Sediment Removal, Green Bay, WI: Project Manager. Project included the design, estimating, procurement, mobilization, demobilization and operations of fast track hydraulic dredging and mechanical dewatering of 50,000 cy of PCB contaminated river sediments under USEPA Emergency Response Program. Project included:

- Construction of an on-site solids separation system consisting of sludge slurry agitated storage tanks, pipelines, vibrating shakers, desanders, and eight recessed-chamber plate and frame filter presses (1,710 cf).
- Design, installation and operation of a separation and water treatment train for a dredge-water flow rate of 2,400 gpm.
- Design, construction and operation of an on-site temporary wastewater treatment system for filtrate, decon and storm water with modu-tanks, clarifiers, secondary settling, pH neutralization, oxygenation, and sand and carbon filtration.
- Placement of 6" sand cap over dredged area after verification analytical results confirmed areas were below targeted criteria.
- Mobilization of an operations plant housing over 125 electrical motors requiring 480 volt 3 phase 2400 amp electrical power Project was completed ahead of schedule in 23 days and mobilization effort completed in 10 days from the notice of contract award to the first day personnel and equipment arrived on-site.

Cumberland Bay, Sludge Removal, Plattsburgh, NY: Dewatering Operations Manager for the design, estimating, procurement, mobilization, staffing, and shake out of the dredging, dewatering, and wastewater treatment systems O+M. Project required hydraulic dredging in excess of 130,000 cy PCB contaminated sludge and lake sediments in three fined areas (57 acres); dewatering of sediments through a bank of plate and frame filter presses (1,760 cf); installation and O+M of a wastewater system to treat approximately 140 million gallons of filtrate; installation of 1,000' steel sheet piling; deployment of 8,000'+ of turbidity curtains; selective shoreline excavation; characterization, transportation and disposal of solids; extensive site restoration. This project was contracted to complete the dredging of 130,000 cubic yards over five years approximately 150,000 yards in the first year using 105 dredging and dewatering operating days. Developed and implemented elaborate electronic and conventional survey control grid systems or the hydraulic dredging in Cumberland Bay and Fox River which resulted in accurate hydraulic dredging of sediment, a reduction in overcuts and significant cost savings in treatment and disposal. Original scope of 60 months was completed in 105 days team yielded 30,000 cy over goal.

Years of Experience:
16

Education:

- BS, Environmental Studies, State University of New York , Buffalo, NY, June 1997

Certifications:

- OSHA 40 Hour HAZWOPER Training
- OSHA 8 Hour HAZWOPER Refresher

Role and Responsibility:

- Responsible for delivery of completed project in compliance with plans and specifications.
- Attends all scheduled progress meetings.
- Meets daily with the project team to review planned activities.
- Develops and maintains all project deliverables, submittals, schedule and reports.
- Develops and manages cost control and accounting, procurement and expediting material delivery, inspection, and quality control functions.

Experience and Expertise:

- Remediation skills developed out of heavy, highway and civil construction project experience.
- Understands CERCLA, RCRA, TSCA and additional regulatory requirements.
- Firm Fixed Price, Cost Reimbursable, Fixed Fee, Guaranteed Maximum Price Contract Experience
- Union and Open Shop Labor
- Dredging, Dewatering and Filtrate Treatment
- Excavation and Earthwork
- Steel Sheeting Systems
- Recovery and Treatment Systems
- Solidification/Stabilization
- Constructability Reviews

Mark Schmitt

Project Manager

Past Project Experience

Menominee River Sediment Removal Project, Marinette, WI: Project Manager. Mechanical environmental dredging of approximately 235,000 cy of river sediment. Dredged sediments loaded onto hopper barges. Barges transport sediments to an offloading and storage area. Stabilization of sediments using a pugmill. Processed sediment for offsite transportation and disposal. Dredge water treated in WWTP prior to discharge.

Bridgeton Former MGP Site, Bridgeton, NJ: Project Manager for the excavation, transportation and disposal of approximately 45,000 CY of impacted soils for thermal treatment or landfill. Final waste disposition was based upon results from sampling and analytical testing. Excavation included deep cuts to 21 ft, cuts and water handling adjacent to the Cohansey River and excavation of sediment from the river. Installation, O+M of a well point dewatering system and 750 gpm temporary groundwater treatment system to manage water during excavations. All water encountered during remediation was collected and treated onsite. Installation of an approximate 270 LF steel sheet pile retaining wall using 65 ft sheets and three rows of tie-backs to support deep excavation at the toe of steep slope.

Ventron/Velsicol Superfund Site OU-1, Bergen County, NJ: Project Manager. Installation of multiple temporary cantilever sheet pile shoring systems to stabilize excavation sidewalls and prevent groundwater infiltration. Performed precise excavations to highly variable, predetermined cut depths managing excavation sequencing to maintain sidewall heights within OSHA limits. Screened oversized debris (>2 inches) from the excavated material for reuse as backfill to minimize offsite disposal costs. Monitored, managed, and contained mercury vapors during excavation and material handling operations. Performed delicate excavation and backfilling operations around the perimeter of the site (buffer zone) that was highly impacted by tidal flow while limiting sediment migration (turbidity levels).

Former MGP Site Remediation, Sag Harbor, NY: Project Engineer for the construction of a temporary water treatment system sized at 1,000 gpm; installation of a network of dewatering wells; construction of a subsurface Soil Mix Wall (1,000 lf x 6-17 ft BGS); management of all SMW spoils; connection of WTP effluent to the offshore pipeline; excavate impacted soil and subsurface structures; transportation of impacted materials to an off-site permitted treatment/disposal facility; and site restoration. Impacted soil excavation, solidification /stabilization, stockpiling and loading performed under a temporary fabric structure.

Former Brooklyn Borough Gas Works Site OU2 &OU3, Brooklyn, NY: Project Engineer. The project required the installation of approximately 2100 linear feet of permanent impermeable sheet piling along the shore of Coney Island Creek as well as the mechanical dredging and ex-situ stabilization of approximately 60,000 cy of contaminated sediment. The dredged sediments were processed inside a temporary onsite soil amendment facility prior to the creek being capped. The creek capping operation included the installation of a geo-membrane fabric and the placement of approximately 62,000 cy of a sand and gravel product along the entire creek. In addition, approximately 55,000 cy of MGP impacted soils were excavated and thermally treated offsite as a result of the former coal tar processing plant operations. A NAPL collection trench and associated wells was also installed. The 20 acre (+/) site was then capped with a gravel layer, clay layer, topsoil and then seeded.

Former MGP Site Remediation, Long Branch, NJ: Project Manager for the remediation of contaminated soils from an inactive MGP site located adjacent to an active railway, residential and church properties. Sevenson excavated, segregated and processed (via solidification) 68,000 cy of material. Treated material was loaded into trucks for offsite T+D. Solidification operations were conducted in a temporary fabric structure. Additional project work tasks included realignment and modifications to an existing stream. This was accomplished in part with the installation of coffer dams and a bypass pumping system. Sevenson also removed sediment from 1,500 linear feet of stream; installed a natural and synthetic layer cap over stream bed to prevent further contaminant migration; constructed a 5 acre multi-layer cap and concrete retaining wall to support capping system; and demolished buildings and structures within cap boundaries including a gas holder and spray pond foundations, piping and appurtances. Odor suppressant foam used during excavation and material handling activities. Site dewatering and water treatment was ongoing. Extensive restoration to stream and properties was required.

Fields Brook Superfund Site, Ashtabula, OH: Project Engineer / QA/QC for remedial action of the sediment and floodplains/wetlands operable unit. Project requires excavation, handling, transport and placement into an on-site landfill 30,000 cy of material removed over 22 miles of creek. Additional tasks include: operation and maintenance of on-site wastewater treatment facility, backfilling and revegetating floodplain areas, restoring stream banks with erosion-resistant materials, installation of landfill cover system and excavation, transport and placement of approximately 10,000 cy of DNAPL material which will be thermally treated on-site.

PCB Sediment Removal, Russelville, KY: Project Engineer for the excavation, characterization, transport and disposal of 66,000 cy of PCB contaminated sediments from creek and flood plain areas along Town Branch Creek. Installation of a 200 gpm wastewater treatment system. Installation of rip rap and complete site restoration.

Steven C. Shaw, P.E.

Environmental Manager

Years of Experience:
10

Education:

- M.Eng., Environmental Engineering, Johns Hopkins (2009)
- B.S., Environmental Systems Engineering, Pennsylvania State University (2002)
- Professional Engineer (Environmental) 2009, DE, MD, NJ, NY, PA, VA,

Certifications and Seminars:

- OSHA 40 Hour HAZWOPER Training
- OSHA 8 Hour HAZWOPER Refresher
- TWIC Approved
- Dredging Engineering Short Course – Texas A & M University (2004)
- GBA/USACE Hydrographic Survey Course (2008)
- Specifying & Interpreting Geotechnical Lab Tests – Virginia Tech. University (2005)
- Deep Foundations Seminar - Marine Foundations (2009)
- Battelle Remediation of Contaminated Sediment

Papers/Publications:

Masonville Dredged Material Containment Facility: Dredging Engineering & Design.

Shaw, S.C., WEDA XXX / TAMU 41 Conference, June 2010.

Paul S. Sarbanes Ecosystem Restoration at Poplar Island: Dike Construction & Wetland Development. Shaw, S.C. & Donegan, T.M WODCON XVII - Global Dredging Conference, May, 2007.

Employment History

2014 to Present	**Sevenson Environmental Services, Inc.**
2004 to 2014	**Gahagan & Bryant Associates, Inc.**

Experience and Expertise

Mr. Shaw has more than 10 years of experience in the areas of civil, environmental, and dredging engineering; containment facility design and dredged material disposal techniques; environmental dredging and processing of sediments; marine construction cost estimating and construction management; port and harbor planning and development; coastal permitting; navigation channel design; dredging production analysis; and dredge/marine equipment selection.

Past Project Experience

Robert Moses State Park, Fire Island, NY: In response to Superstorm Sandy, Mr. Shaw managed an internal team through an emergency dredging and truck haul contract for the renourishment of Robert Moses State Park Beach and maintenance dredging of the State Boat Ramp Channel. Within 5 days of client contact, the survey, design, contract documents, and bid package was prepared for advertisement. The final project (~$8 Million) included 630,000 cy of beach fill (400,000 cy hydraulically dredged and placed and 230,000 cy stockpiled and hauled), grading/shaping, Piping Plover mitigation, and numerous owner requested contract modifications.

Delaware River Main Channel Deepening, PA, NJ, DE: Mr. Shaw was the engineering and technical lead responsible for working on behalf of the local sponsor with the USACE to deepen the Delaware River Main Channel from 40 ft MLLW to 45 ft MLLW. The 16 Mcy, $300Million, 103-mile federally authorized project encompasses mechanical, cutter-suction, and hopper dredging; rock drilling and blasting; wetland restoration; beach nourishment; CDF disposal management; and multiple contracts. Mr. Shaw oversaw the geotechnical data collection, scheduling, contracting, plans and specifications, construction management, dredging inspection, and coordination with both the local sponsor and the USACE.

***NYC Combined Sewer Outfalls (CSO), New York City, NY*:** Mr. Shaw provided engineering design services for multiple CSO dredging projects, ranging from $10 to $50 Million each, in the New York City area. The work includes hydrographic surveys, side scan surveys, and magnetometer surveys for remedial investigations; production and cost estimation; selection of remediation techniques and dredging methods, and contract plans and specifications for final remedial design. Mr. Shaw was responsible for work products associated with the remediation projects and responsible for the selection of remediation techniques and

dredging methods, dredge/processing production, and engineer's cost estimates.

Hart-Miller Island DMCF Management & Closure, Baltimore, MD: Hart Miller Island is a 1,100 acre diked dredged material containment area with a capacity of 100 Mcy, in the Chesapeake Bay near the Port of Baltimore. State legislature required its closure by December 31, 2009. Mr. Shaw was responsible for preparation of the final hydraulic filling plan to maximize the remaining capacity of the site, while also accommodating the site's closure plan elevations.

Innovative Re-Use Program, MD: The Maryland Port Administration continues to actively fund and investigate methods and methodologies designed to innovatively reuse dredged sediments at their Cox Creek DMCF. Mr. Shaw was managing the technical, financial, and operational review and analysis of a private proposal to manufacture light weigh aggregate (LWA) from dredged sediments.

Masonville DMCF Construction, Baltimore, MD: To meet the client's annual dredged material placement demand (1.5 Mcy/yr), the in-water 141-acre Masonville DMCF was constructed within the Baltimore Harbor. To reduce construction costs and increase site capacity, the dikes were constructed with 800,000 cy of materials (sand, gravel, clay) hydraulically excavated from within the project footprint and pumped into place, saving the client approximately $7.5M. Mr. Shaw participated as the project manager responsible for contract oversight, construction inspection, daily contractor and owner reporting, progress and payment surveys, coordination with permitting agencies, owner/contractor liaison, and field crew management.

Masonville DMCF Pre-Dredging & Seagirt/Dundalk Channel Deepening, Baltimore, MD: Two unrelated projects were combined into one intertwining contract ($42 M, 4.6 Mcy) to reduce mobilization costs and to beneficially reuse suitable material from the deepening project as construction material on the DMCF pre-dredging contract, saving the owner nearly $10M. Mr. Shaw played an integral part in developing the design, plans, and specifications for the project which included template and performance dredging, hydraulic and mechanical placement, and bottom dumping of dredged materials. During Construction, Mr. Shaw was the project engineer responsible for contract oversight, construction inspection, daily reporting, weekly summaries, and field crew management.

Portland Harbor Superfund, Willamette River Portland, OR: Mr. Shaw developed engineer’s cost estimates for various dredging scenarios and methods, utilizing several sediment suitability ranking methods and placement alternatives. The scenarios developed by Mr. Shaw required the selection and sizing of dredging equipment, production estimates and comparisons, consideration of environmental windows and constraints, and analysis of sediment data (both chemical and physical). The project area includes over 10 miles of a federal navigation channel and quantities ranged from 4 to 8 Mcy.

Market Study - Dredged Material Placement/Rehandling: Mr. Shaw performed a study to analyze the market conditions and feasibility for developing a privately owned site into a facility to accept dredged material. The study included a comprehensive review of the site conditions to assess the feasibility and construction costs of developing a rehandling basin for the bottom-dumping of scow transported dredged material and a financial analysis to assess potential tipping fees and rate of return.

***Paulsboro Marine Terminal Design & Construction, Paulsboro, NJ*:** Project manager responsible for the design, permitting, dredged material management plan, engineer's cost estimate, construction plans, and specifications for the dredging and dredged material related activities associated with the $135M marine terminal development of the new Port of Paulsboro. During construction, Mr. Shaw also managed the construction management, inspection and hydrographic surveying services for the owner. The project included the dredging of 320,000 cy. A total of 240,000 cy were beneficially used onsite to cap and increase terminal elevations and the remaining 40,000 cy required amending with Portland cement and offsite placement.

Dredged Material Management Program, Baltimore, MD: Mr. Shaw was the project manager responsible for ongoing dredged material management plan technical services for Maryland Port Administration (MPA). Services for this $50 M/yr program to manage 5.4 Mcy of dredged material are varied and are of an immediate and timely nature, requiring an intimate working knowledge of the USACE - Baltimore, Norfolk, and Philadelphia District and the MPA Dredged Material Management Program needs; the Chesapeake Bay local, state and federal agency authorization and appropriation processes; state and federal (NEPA) permitting; and the USACE Civil Works Construction Programs.



Nicole La Plant

Senior Project Geologist

EXPERTISE:

- 14 years experience in environmental consulting covering the following areas of expertise:
- Property Transaction Environmental Due Diligence
- Brownfield Redevelopment
- Municipal Water Supply Well Siting and Development
- Site Investigation and remedial activities
- Monitoring well installation, development, and groundwater sampling
- Drilling, remedial excavations, and spill response field activity oversight

CONTINUING EDUCATION:

- 40-hour OSHA Hazardous Site Safety Training 29 CFR 1910-120
- State of WI Certified Asbestos Inspector (#All-111258)
- State of WI Certified Tank System Site Assessor (#46836)

EDUCATION:

- University of WI—Green Bay
  B.S. Urban and Public Affairs
- University of WI—Oshkosh
  Geology Course Work To Meet Ch. NR 712, WI Administrative Code

REGISTRATION:

- Registered Petroleum Environmental Cleanup Fund Act (PECFA) Consultant

PROFESSIONAL ORGANIZATION:

- WI Groundwater Association

Ms. La Plant is a senior project geologist at Robert E. Lee & Associates, Inc. and specializes in the implementation and management of project work plans. Her qualifications are as follows:

- Completion of asbestos inspections for various residential and commercial buildings.
- Knowledge of environmental regulations and guidelines.
- Borehole logging, screening, and sampling.
- Monitoring well installation, development, and sampling groundwater.
- Methane gas probe installation and monitoring techniques.
- Land surveying methods and techniques.
- Preparation of project work plans and performing site scoping.
- Evaluation of geologic, hydrogeologic, and contaminant distribution data.
- Evaluation of selection of appropriate remedial alternatives.
- Technical report preparation and documentation.
- Preparation and submission of PECFA claims and Dry Cleaning Environmental Response Fund (DERF) program applications.
- Completion of Phase I and Phase II environmental site assessments of commercial and private properties throughout Wisconsin and Michigan.
- Petroleum hydrocarbon, chlorinated solvent, agricultural chemical and polychlorinated biphenyl (PCB) investigation and remediation.
- Oversee field activities associated with drilling, remedial excavations, and emergency spill response cleanup.
- Preparation of Quality Assurance Project Plan (QAPP) for U.S Environmental Protection Agency Brownfields Assessment Grant project.



Jared G. Schmidt, P.E.
Principal
Civil/Municipal Engineering Manager

EXPERTISE:
- Civil/Municipal Engineering Manager
- Street, sanitary sewer, water main, and storm sewer design
- Site plan development and design
- Storm water management design and reporting
- Hydraulic and hydrologic studies and analysis
- Flood plain and dam break analysis
- Agency permitting, local, state, and federal

EDUCATION:
- Marquette University, Milwaukee WI
  B.S., Civil Engineering, 2003

REGISTRATION:
- Professional Engineer, WI

PROFESSIONAL ORGANIZATION:
- American Society of Civil Engineers
- Fox Wolf Watershed Alliance ~ Vice President
- FWWA Conferences ~ Planning team
- Northeast Wisconsin Storm Water Consortium ~ Member
- Brown County Home Builders Association ~ Associate Director

Site and Storm Water Plan Design (Various Projects within WI). Project Engineer for various site plan designs throughout Wisconsin. Design responsibilities include layouts of physical improvements including building location, parking lots, and green space. Design includes detailed utility layout (water main, sanitary & storm sewer), grading plan, and erosion control plan. Complete storm water Best Management Practices design, reporting, and permitting are typically required for this work. Project examples include:

Bellin Hospital Courtyard Addition, Green Bay, WI.
Bellevue Crossing, Bellevue, WI.
Globe University, Bellevue & Grand Chute, WI.
Jones Sign, Lawrence, WI.
South Bay Marina, Green Bay, WI.
EMT International, Hobart, WI.
Centennial Estates, Hobart, WI.
Schindlers Place Estates, Appleton, WI.
KI, Inc., Bellevue, WI.

Storm Water Management Design (Various Projects within WI). These regional facility designs included the development of regional storm water Best Management Practices (BMPs) for previously or newly developed areas. Responsibilities include identification of complete drainage basins, land uses, storm sewer conveyance systems, and site soils to design site specific BMPs. Regional ponds or other BMPs are then designed and evaluated for storm water quality and quantity impact. Project examples include:

"The Woods" Golf Course Storm Water Pond, Green Bay, WI.
Brown County Farms Pond, Green Bay, WI.
Centennial Centre at Hobart Regional Storm Plan, Hobart, WI.
Fochs Trails, Hilbert, WI.
Rockville Heights, Kiel, WI.
Glen Kent Estates, Howard, WI.
Pulaski School District Pond Retrofit, Pulaski, WI.

Waterway Analysis & Design (Various Projects within WI). Jared has been involved in the hydraulic & hydrologic analysis of numerous waterways throughout the state. Many projects started out by analyzing the existing 100-year flood plain elevation and location. This data can be used for FEMA flood plain mapping; as a base line for future development, properly sizing culverts, or for stream restoration. By utilizing HEC-RAS, detail analysis and design can be performed. Many waterway projects also involve close coordination with permitting agencies, including local, state and federal. Other project examples include:

Mosher Creek Stream Restoration, North Fond Du Lac, WI.
Lane Creek Stream Restoration, Town of Patch, Grant County, WI.
Porlier Pier Permitting, Green Bay, WI.
Vine Road Box Culvert Replacement, Freedom, WI.
Ashwaubenon Creek Flood Plain Analysis, Lawrence, WI.
East River Flood Plain Analysis, Wrightstown, WI.

Jared G. Schmidt, P.E.

CONTINUING EDUCATION:

- Dam break analysis using HEC-RAS
- Source Loading and Management Model (SLAMM)
- P8 urban catchment model
- Post-construction storm water management work shop
- Construction erosion control workshop
- Phase II storm water regulation and compliance
- Docks and marinas

Centennial Centre at Hobart, Municipal Engineering Project, Village of Hobart, Brown County, WI. Project design/project manager for the construction of 12,500 lineal feet of 8"-18" sanitary sewer, 12,000 lf of 8"-12" water mains, 10,000 lf of 12"-36" storm sewer, corresponding laterals for servicing a multi-use development. Design also includes a four-lane boulevard, concrete roadways, a round-a-bout with colored and stamped concrete. Other critical elements of this development include design of a regional storm water management plan, detailed streetscaping, and selection and design of a private decorative lighting system. Other project examples include:

City of Kiel East Industrial Park, Kiel WI.
North Avenue Road, Denmark, WI.
TID Planning and Creation, Hobart, WI.
Crivitz Drainage Improvements, Crivitz, WI.
Cobblestone Estates, Hobart, WI.
Tailwind Crossing, Hobart, WI.

Shawano Avenue, Street and Utility Design, City of Green Bay, Brown County, WI. Design engineer for the reconstruction of Shawano Avenue in the City of Green Bay Reconstruction extended from Ridge Road to Military Avenue. Design elements included narrowing of the road to help reduce travel speeds, increase terrace widths for increased snow removal storage, and the elevating of the roadway to accommodate a former rail crossing and add an at-grade, multi-use trail.

Recreational Design and Planning. Project Manager and Design Engineer for multiple recreational projects and athletic field planning and design. Includes spatial layout of ball diamonds, soccer fields, shot put, and discuss areas. Planning also includes placement of parking areas, bleachers, concession stands, lighting, and multi-use recreation trails. Project examples include:

Village of Pulaski - Memorial Park Ball Diamonds.
Denmark School District - Soccer and Track Areas.
Hobart, Sister Bay, Waupaca Multi-Use Trails.

APPENDIX I

Sample Tables include:

Table 5. Compound List, Quantitation Limits, and Standards – PACE Laboratories

Table 6. Field and Lab QA/QC Sample Requirements

Table 5. Compound List, Quantitation Limits, and Standards - PACE Laboratories

ICP Metals (mg/Kg)
Soil

Analytes	Analytical Method	Current Reporting Limits	Current LOQ	NR 720 RCL (mg/Kg)		MS/MSD %R	MS/MSD %RPD
				Non-Industrial	Industrial		
Aluminum	6010C	8.7434	50.00			75-125	20
Antimony	6010C	0.5676	2.00			75-125	20
Arsenic (high level)	6010C	0.6365	2.00	0.039	1.6	75-125	20
Arsenic	7010	-	-	0.039	1.6	-	-
Barium	6010C	0.1193	0.50	ns	ns	75-125	20
Beryllium	6010C	0.0376	0.40			75-125	20
Cadmium	6010C	0.0662	0.50	8	510	75-125	20
Calcium	6010C	2.7385	100.00			75-125	20
Chromium Hexavalent	7196A	-	-	14	200	-	-
Chromium Trivalent	6010C	-	-	16000	N/A	N/A	N/A
Chromium	6010C	0.1939	0.50			75-125	20
Cobalt	6010C	0.0972	0.50			75-125	20
Copper	6010C	0.1561	1.00			75-125	20
Iron	6010C	1.6801	10.00	ns	ns	75-125	20
Lead	6010C	0.4308	1.00	50	500	75-125	20
Magnesium	6010C	5.4229	100.00			75-125	20
Manganese	6010C	0.0508	0.50			75-125	20
Nickel	6010C	0.1295	1.00			75-125	20
Potassium	6010C	8.222	100.00			75-125	20
Selenium	6010C	0.7709	2.00	ns	ns	75-125	20
Silver	6010C	0.2782	1.00	ns	ns	75-125	20
Sodium	6010C	3.8451	100.00			75-125	20
Thallium	6010C	0.8199	4.00			75-125	20
Vanadium	6010C	0.2043	1.00			75-125	20
Zinc	6010C	0.3851	4.00			75-125	20

Mercury (mg/Kg)
Soil

Analytes	Analytical Method	Current Reporting Limits	Current LOQ	NR 720 RCL (mg/Kg)		MS/MSD %R	MS/MSD %RPD
				ES	PAL		
Mercury	7471	0.0033	0.0067	0.0031	ns	85-115	20

Cyanide (mg/Kg)
Soil

Analytes	Analytical Method	Current Reporting Limits	Current LOQ	NR 720 RCL (mg/Kg)		MS/MSD %R	MS/MSD %RPD
				ES	PAL		
Cyanide	9012A	0.08722	0.60	ns	ns	80-120	20

RCL NR 720 Wisconsin Administrative Code Residual Contaminant Level (RCL)
ns No NR 720 Wisconsin Administrative Code Residual Contaminant Level (RCL) established
mg/Kg Standards reported as milligrams per kilogram, equivalent to parts per million (ppm)

PAH 8270C SIM (mg/Kg)
Soil

Analytes	Current Reporting Limits	Current LOQ	Suggested Generic RCLs (mg/Kg)			MS/MSD %R	MS/MSD %RPD
			Ground water Pathway	Direct Contact Pathway			
				Non-Industrial	Industrial		
1-Methylnaphthalene	0.008333	0.01667	23	1100	70000	42-130	32
2-Methylnaphthalene	0.008333	0.01667	20	600	40000	34-130	35
Acenaphthene	0.008333	0.01667	38	900	60000	31-130	35
Acenaphthylene	0.007457	0.01667	0.7	18	360	32-130	25
Anthracene	0.008641	0.01667	3000	5000	300000	39-131	38
Benzo(a)anthracene	0.005777	0.01667	17	0.088	3.9	29-130	30
Benzo(a)pyrene	0.005959	0.01667	48	0.0088	0.39	35-130	33
Benzo(b)fluoranthene	0.008333	0.01667	360	0.088	3.9	21-142	44
Benzo(g,h,i)perylene	0.006348	0.01667	6800	1.8	39	12-134	33
Benzo(k)fluoranthene	0.009223	0.01667	870	0.88	39	35-130	37
Chrysene	0.007706	0.01667	37	8.8	390	37-130	38
Dibenzo(a,h)anthracene	0.006113	0.01667	38	0.0088	0.39	23-130	27
Fluoranthene	0.008333	0.01667	500	600	40000	29-137	50
Fluorene	0.008333	0.01667	100	600	40000	32-130	32
Indeno(1,2,3-cd)pyrene	0.006332	0.01667	680	0.088	3.9	17-134	28
Naphthalene	0.008333	0.01667	0.4	20	110	24-130	40
Phenanthrene	0.008333	0.01667	1.8	18	390	27-135	46
Pyrene	0.008333	0.01667	8700	500	30000	24-130	49

Note: Soil standards listed for PAHs are “suggested generic residual contaminant levels” outlined in WDNR Guidance Publication (RR-519-97) Soil Cleanup Levels for Polycyclic Aromatic Hydrocarbons Interim Guidance (1997). This guidance does not establish or affect legal rights or obligations.

Table 5 (continued)

SVOC 8270 (mg/Kg)
Soil

Analytes	Current Reporting Limits	Current LOQ	NR 720 RCL	USEPA RSLs	MS/MSD %R	MS/MSD %RPD
1,2,4,5-Tetrachlorobenzene	0.06069	0.167	ns	18	47-130	26
1,2,4-Trichlorobenzene	0.04174	0.167	ns	22	47-130	26
1,2-Dichlorobenzene	0.02767	0.167	ns	1900	49-130	33
1,2-Diphenylhydrazine	0.04742	0.167	ns	0.61	10-150	42
1,3-Dichlorobenzene	0.02213	0.167	ns	530	46-130	34
1,4-Dichlorobenzene	0.02508	0.167	ns	2.4	48-130	34
2,4,5-Trichlorophenol	0.03547	0.167	ns	6100	16-130	21
2,4,6-Trichlorophenol	0.04226	0.167	ns	44	11-132	33
2,4-Dichlorophenol	0.02780	0.167	ns	180	32-130	31
2,4-Dimethylphenol	0.03193	0.167	ns	1200	17-139	29
2,4-Dinitrophenol	0.06769	0.333	ns	120	10-130	40
2,4-Dinitrotoluene	0.05140	0.167	ns	16	22-146	42
2,6-Dichlorophenol	-	-	ns	ns	-	-
2,6-Dinitrotoluene	0.01816	0.167	ns	61	30-136	38
2-Chloronaphthalene	0.03038	0.167	ns	6300	39-130	25
2-Chlorophenol	0.03008	0.167	ns	390	39-130	31
2-Methylnaphthalene	0.03092	0.167	ns	310	37-130	29
2-Methylphenol	0.03165	0.167	ns	3100	33-130	29
2-Nitroaniline	0.03136	0.167	ns	610	33-135	33
2-Nitrophenol	0.03259	0.167	ns	ns	22-130	41
3 & 4-Methylphenol	0.02438	0.167	ns	310	28-130	29
3,3'-Dichlorobenzidine	0.02061	0.167	ns	1.1	10-173	50
3-Nitroaniline	0.04603	0.167	ns	18	10-166	40
4,6-Dinitro-2-methylphenol	0.01752	0.167	ns	4.9	10-130	50
4-Bromophenyl-phenyl ether	0.03510	0.167	ns	ns	27-130	31
4-Chloro-3-methylphenol	0.03235	0.167	ns	6100	29-130	31
4-Chloroaniline	0.02072	0.333	ns	2.4	20-132	36
4-Chlorophenyl-phenyl ether	0.03488	0.167	ns	ns	35-130	34
4-Nitroaniline	0.04389	0.167	ns	24	10-178	37
4-Nitrophenol	0.04478	0.167	ns	ns	10-142	47
Acenaphthene	0.03981	0.167	ns	3400	57-130	34
Acenaphthylene	0.03724	0.167	ns	ns	38-130	34
Acetophenone	0.21524	0.333	ns	7800	10-150	40
Aniline	0.03316	0.167	ns	85	10-149	34
Anthracene	0.04403	0.167	ns	17000	29-130	38
Benzidine	0.19240	3.333	ns	0.0005	10-150	40
Benzo(a)anthracene	0.03017	0.167	ns	0.15	19-132	42
Benzo(a)pyrene	0.03314	0.167	ns	0.015	11-131	38
Benzo(b)fluoranthene	0.03943	0.167	ns	0.15	10-133	40
Benzo(g,h,i)perylene	0.03343	0.167	ns	ns	14-130	47
Benzo(k)fluoranthene	0.04520	0.167	ns	1.5	18-136	42
Benzyl alcohol	0.04516	0.333	ns	6100	36-130	35

Bis(2-chloroethoxy)methane	0.05250	0.167	ns	180	55-130	22
Bis(2-chloroethyl)ether	0.04675	0.167	ns	0.21	47-130	24
Bis(2-chloroisopropyl)ether	0.02889	0.167	ns	2.9	43-130	26
Bis(2-ethylhexyl)phthalate	0.28429	3.333	ns	35	10-163	34
Butylbenzylphthalate	0.02954	0.167	ns	260	10-163	39
Carbazole	0.02108	0.167	ns	1500	16-144	32
Chrysene	0.03131	0.167	ns	15	25-130	41
Dibenzo(a,h)anthracene	0.03283	0.167	ns	0.015	11-130	38
Dibenzofuran	0.02457	0.167	ns	78	32-130	35
Diethylphthalate	0.03668	0.167	ns	49000	27-134	36
Dimethylphthalate	0.03306	0.167	ns	1000000	30-131	37
Di-n-butylphthalate	0.03968	3.333	ns	6100	19-142	33
Di-n-octylphthalate	0.03593	0.167	ns	2400	10-160	42
Fluoranthene	0.03192	0.167	ns	2300	20-143	40
Fluorene	0.03584	0.167	ns	2300	36-130	32
Hexachlorobenzene	0.03683	0.167	ns	0.3	28-130	34
Hexachlorobutadiene	0.02595	0.167	ns	6.2	40-130	21
Hexachlorocyclopentadiene	0.03075	0.167	ns	370	10-130	45
Hexachloroethane	0.03766	0.167	ns	12	32-130	37
Hexachloropropene	-	-	ns	ns	-	-
Indeno(1,2,3-cd)pyrene	0.02661	0.167	ns	0.15	10-130	48
Isophorone	0.01725	0.167	ns	510	49-130	20
Naphthalene	0.03779	0.167	ns	3.6	55-130	31
Nitrobenzene	0.03770	0.167	ns	4.8	45-130	30
N-Nitrosodimethylamine	0.03103	0.167	ns	0.0023	37-147	29
N-Nitroso-di-n-propylamine	0.02948	0.167	ns	0.069	46-130	27
N-Nitrosodiphenylamine	0.04075	0.167	ns	99	47-130	26
N-Nitrosopyrrolidine	-	-	ns	0.23	-	-
Pentachlorophenol	0.02520	0.167	ns	0.89	10-130	50
Phenanthrene	0.03561	0.167	ns	ns	26-131	35
Phenol	0.12653	0.333	ns	18000	39-130	30
Pyrene	0.02254	0.167	ns	1700	10-155	44
Pyridine	0.03497	0.167	ns	78	10-130	40

RCL NR 720 Wisconsin Administrative Code Residual Contaminant Level (RCL)
ns No NR 720 Wisconsin Administrative Code Residual Contaminant Level (RCL) established
mg/Kg Standards reported as milligrams per kilogram, equivalent to parts per million (ppm)
RSL USEPA Region 9 Regional Screening Level (November 2011). RSLs listed are based on residential direct contact pathway. The RSLs contained in the table are generic, and should be viewed as screening guidelines, not legally enforceable standards.

Solids by 9017B
Soil

Analytes	MDL (mg/Kg)	PRL (mg/Kg)	LCS/LCSD			MS/MSD %R			DUP
			Lower	Upper	RPD	Lower	Upper	RPD	RPD
Oil & Grease	63.8	250	78	114	18	78	114	18	18
TPH	72.9	250	64	132	34	64	132	34	34

Table 5 (continued)

ICP Dissolved (µg/L) - Groundwater

Analytes	Analytical Method	Current Reporting Limits	Current LOQ	NR 140 Groundwater Standards		MS/MSD %R	MS/MSD %RPD
				ES (µg/L)	PAL (µg/L)		
Aluminum	6010C	65.5036	500.00	200	40	75-125	20
Antimony	6010C	7.2633	20.00	6	1.2	75-125	20
Barium	6010C	1.6987	5.00	2 mg/L	0.4 mg/L	75-125	20
Beryllium	6010C	0.6826	4.00	4	0.4	75-125	20
Cadmium	6010C	0.9974	5.00	5	0.5	75-125	20
Calcium	6010C	23.5272	1000.00	250 mg/L	125 mg/L	75-125	20
Chromium	6010C	1.5245	5.00	100	10	75-125	20
Cobalt	6010C	1.2957	5.00	40	8	75-125	20
Copper	6010C	3.3763	10.00	1300	130	75-125	20
Iron	6010C	15.9204	100.00	0.3 mg/L	0.15 mg/L	75-125	20
Lead	6010C	1.6164	7.50	15	1.5	75-125	20
Magnesium	6010C	66.9159	1000.00	0.05 mg/L	0.025 mg/L	75-125	20
Manganese	6010C	0.3697	5.00	300	60	75-125	20
Nickel	6010C	1.2635	10.00	100	20	75-125	20
Potassium	6010C	64.8712	1000.00	ns	ns	75-125	20
Selenium	6010C	6.8469	20.00	50	10	75-125	20
Silver	6010C	3.1619	10.00	50	10	75-125	20
Sodium	6010C	46.2787	1000.00	ns	ns	75-125	20
Thallium	6010C	7.081	40.00	2	0.4	75-125	20
Vanadium	6010C	2.4789	10.00	30	6	75-125	20
Zinc	6010C	5.757	40.00	5 mg/L	2.5 mg/L	75-125	20

Arsenic Dissolved (µg/L) - Groundwater

Analytes	Analytical Method	Current Reporting Limits	Current LOQ	NR 140 Groundwater Standards		MS/MSD %R	MS/MSD %RPD
				ES (µg/L)	PAL (µg/L)		
Arsenic	6010C	6.8149	20.00	10	1	75-125	20

Table 5 (continued)

Hexavalent Chromium Dissolved (µg/L) - Groundwater

Analytes	Analytical Method	Current Reporting Limits	Current LOQ	NR 140 Groundwater Standards		MS/MSD %R	MS/MSD %RPD
				ES (µg/L)	PAL (µg/L)		
Chromium $^{+6}$	3500 Cr-D	3.9	20.0	100	10	90-110	20

Mercury Dissolved (µg/L) - Groundwater

Analytes	Analytical Method	Current Reporting Limits	Current LOQ	NR 140 Groundwater Standards		MS/MSD %R	MS/MSD %RPD
				ES (µg/L)	PAL (µg/L)		
Mercury	7470	0.100	0.200	2	0.2	-	-

Cyanide Dissolved (µg/L) – Groundwater

Analytes	Analytical Method	Current Reporting Limits	Current LOQ	NR 140 Groundwater Standards		MS/MSD %R	MS/MSD %RPD
				ES (µg/L)	PAL (µg/L)		
Cyanide	9012A	10.00	20.00	200	40	90-110	20

ES Enforcement Standard established in NR 140, Wisconsin Administrative Code
PAL Preventive Action Limit established in NR 140, Wisconsin Administrative Code
ns No NR 140 Wisconsin Administrative Code groundwater established for this parameter
µg/L Standards reported as micrograms per liter, equivalent to parts per billion (ppb), except as noted

Table 5 (continued)

PAH 8270C SIM HVI (µg/L)
Groundwater

Analytes	Current Reporting Limits	Current LOQ	NR 140 Groundwater Standards		MS/MSD %R	MS/MSD %RPD
			ES (µg/L)	PAL (µg/L)		
1-Methylnaphthalene	0.005540	0.05	ns	ns	10-130	49
2-Methylnaphthalene	0.00641	0.05	ns	ns	20-130	50
Acenaphthene	0.00565	0.05	ns	ns	23-130	34
Acenaphthylene	0.00506	0.05	ns	ns	31-130	37
Anthracene	0.00576	0.05	3000	600	21-130	35
Benzo(a)anthracene	0.0535	0.05	ns	ns	10-166	27
Benzo(a)pyrene	0.00398	0.05	0.2	0.02	10-138	28
Benzo(b)fluoranthene	0.00678	0.05	0.2	0.02	10-149	27
Benzo(g,h,i)perylene	0.01339	0.05	ns	ns	11-151	29
Benzo(k)fluoranthene	0.00481	0.05	ns	ns	17-138	26
Chrysene	0.00856	0.05	0.2	0.02	20-134	26
Dibenzo(a,h)anthracene	0.01472	0.05	ns	ns	17-156	30
Fluoranthene	0.00380	0.05	400	80	40-130	26
Fluorene	0.00583	0.05	400	80	30-130	34
Indeno(1,2,3-cd)pyrene	0.01345	0.05	ns	ns	12-157	29
Naphthalene	0.01054	0.05	100	10	24-130	40
Phenanthrene	0.00521	0.05	ns	ns	28-130	30
Pyrene	0.00420	0.05	250	50	16-145	25

Table 5 (continued)

SVOC 8270 (µg/L)
Groundwater

Analytes	Current Reporting Limits	Current LOQ	NR 140 Groundwater Standards		MS/MSD %R	MS/MSD %RPD
			ES (µg/L)	PAL (µg/L)		
1,2,4,5-Tetrachlorobenzene	1.13	5	ns	ns	56-130	28
1,2,4-Trichlorobenzene	1.46	5	70	14	56-130	28
1,2-Dichlorobenzene	1.84	5	600	60	52-130	31
1,2-Diphenylhydrazine	1.29	5	ns	ns	66-130	20
1,3-Dichlorobenzene	1.65	5	600	120	47-130	32
1,4-Dichlorobenzene	1.94	5	75	15	50-130	30
2,4,5-Trichlorophenol	0.76	5	ns	ns	65-130	22
2,4,6-Trichlorophenol	1.05	5	ns	ns	64-130	20
2,4-Dichlorophenol	1.19	5	ns	ns	60-130	27
2,4-Dimethylphenol	0.93	5	ns	ns	10-135	41
2,4-Dinitrophenol	0.86	10	ns	ns	12-162	46
2,4-Dinitrotoluene	1.00	5	0.05	0.005	49-136	26
2,6-Dichlorophenol	-	-	ns	ns	-	-
2,6-Dinitrotoluene	1.55	5	0.05	0.005	56-130	20
2-Chloronaphthalene	1.06	5	ns	ns	65-130	24
2-Chlorophenol	1.03	5	ns	ns	47-130	41
2-Methylnaphthalene	1.66	5	ns	ns	60-130	24
2-Methylphenol	0.96	5	ns	ns	33-130	39
2-Naphthylamine	-	-	ns	ns	-	-
2-Nitroaniline	1.28	5	ns	ns	57-130	23
2-Nitrophenol	0.85	5	ns	ns	53-130	21
3 & 4-Methylphenol	1.28	5	ns	ns	35-130	39
3,3'-Dichlorobenzidine	1.35	5	ns	ns	10-155	44
3-Nitroaniline	1.27	5	ns	ns	32-132	25
4,6-Dinitro-2-methylphenol	0.62	5	ns	ns	15-150	33
4-Bromophenyl-phenyl ether	0.50	5	ns	ns	61-130	21
4-Chloro-3-methylphenol	1.43	5	ns	ns	51-130	33
4-Chloroaniline	1.56	5	ns	ns	32-130	46
4-Chlorophenyl-phenyl ether	0.94	5	ns	ns	66-130	25
4-Nitroaniline	2.23	5	ns	ns	22-153	31
4-Nitrophenol	0.59	10	ns	ns	10-130	25
Acenaphthene	1.34	5	ns	ns	69-130	24
Acenaphthylene	0.94	5	ns	ns	70-130	24
Acetophenone	3.78	10	ns	ns	70-130	20
Aniline	1.70	5	ns	ns	10-134	30
Anthracene	0.56	5	3000	600	69-130	20
Benzidine	28.10	50	ns	ns	10-130	20
Benzo(a)anthracene	0.91	5	ns	ns	64-130	20
Benzo(a)pyrene	1.29	5	0.2	0.02	48-130	27
Benzo(b)fluoranthene	0.70	5	0.2	0.02	50-130	26
Benzo(g,h,i)perylene	2.12	5	ns	ns	48-130	20

Benzo(k)fluoranthene	1.44	5	ns	ns	53-130	28
Benzyl alcohol	1.03	10	ns	ns	39-130	20
Bis(2-chloroethoxy)methane	1.01	5	ns	ns	67-130	20
Bis(2-chloroethyl)ether	0.74	5	ns	ns	59-130	20
Bis(2-chloroisopropyl)ether	1.13	5	ns	ns	55-130	24
Bis(2-ethylhexyl)phthalate	0.77	5	ns	ns	36-143	22
Butylbenzylphthalate	0.76	5	ns	ns	46-130	24
Carbazole	1.02	5	ns	ns	69-130	20
Chrysene	1.04	5	0.2	0.02	68-130	20
Dibenzo(a,h)anthracene	1.00	5	ns	Ns	46-130	22
Dibenzofuran	1.02	5	ns	Ns	70-130	25
Diethylphthalate	0.54	5	ns	ns	59-130	28
Dimethylphthalate	0.73	5	ns	ns	67-130	21
Di-n-butylphthalate	0.95	5	1000	100	59-130	24
Di-n-octylphthalate	1.44	5	ns	Ns	36-147	20
Fluoranthene	1.37	5	400	80	52-130	20
Fluorene	0.88	5	400	80	70-130	20
Hexachlorobenzene	1.82	10	1	0.1	57-130	24
Hexachlorobutadiene	0.57	5	ns	ns	48-130	31
Hexachlorocyclopentadiene	0.90	5	ns	ns	10-130	31
Hexachloroethane	1.48	5	ns	ns	45-130	34
Hexachloropropene	-	-	ns	ns	-	-
Indeno(1,2,3-cd)pyrene	2.10	5	ns	ns	42-130	22
Isophorone	1.03	5	ns	ns	55-130	20
Naphthalene	1.50	5	100	10	60-130	26
Nitrobenzene	1.03	5	ns	ns	62-130	20
N-Nitrosodimethylamine	1.13	5	ns	ns	39-130	20
N-Nitroso-di-n-propylamine	1.02	5	ns	ns	61-130	20
N-Nitrosodiphenylamine	2.18	10	7	0.7	60-150	32
N-Nitrosopyrrolidine	-	-	ns	ns	-	-
Pentachlorophenol	0.75	10	1	0.1	10-149	32
Phenanthrene	0.53	5	ns	ns	68-130	20
Phenol	0.54	5	2	0.4	22-130	33
Pyrene	0.74	5	250	50	58-130	25
Pyridine	1.46	10	10	2	10-130	20

Phenols EPA 420.4 (µg/L)
Water

Analytes	MDL (µg/L)	PRL (µg/L)	LCS/LCSD			MS/MSD %R			DUP
			Lower	Upper	RPD	Lower	Upper	RPD	RPD
Phenols	15.7	50.0	90	110	20	90	110	20	20

Table 5 (continued)

Water 1664A
Liquid/Liquid Hand – Shake Method

Analytes	MDL (µg/L)	PRL (µg/L)	LCS/LCSD			MS/MSD %R			DUP
			Lower	Upper	RPD	Lower	Upper	RPD	RPD
Oil & Grease	0.877	5	78	114	18	78	114	18	18
TPH	1.360	5	64	132	34	64	132	34	34

Water 1664A
SPE/Hotplate

Analytes	MDL (µg/L)	PRL (µg/L)	LCS/LCSD			MS/MSD %R			DUP
			Lower	Upper	RPD	Lower	Upper	RPD	RPD
Oil & Grease	0.877	5	78	114	18	78	114	18	18
TPH	1.360	5	64	132	34	64	132	34	34

Table 6
Field and Lab QA/QC Sample Requirements

	QC Sample Type	Frequency of Sample/Analysis	Details
Field Samples.	Duplicate Samples	1 duplicate per 20 samples per matrix, or 1 duplicate per sample matrix if fewer than 20 samples	Duplicate sample to be collected by the same methods at the same time as the original sample. Used to verify sample and analytical reproducibility.
	Equipment Blanks (or Field Blanks**)	1 equipment blank per 20 samples, minimum 1 equipment blank per day or 1 field blank per bottle lot used, or one per site, whichever is more frequent	Distilled water placed into contact with sampling equipment. Used to assess quality of data from field sampling and decontamination procedures. ** If all disposable equipment/single use sampling equipment is being used, then field blanks may be collected at a rate of 1 per bottle lot or per site, whichever is more frequent.
Lab Samples	Matrix Spike/ Matrix Spike Duplicate	1 MS/MSD per 20 or fewer samples per matrix	Laboratory spiked sample to evaluate matrix and measurement methodology.
	Method Blanks	1 method blank per batch of samples prepared, or per lab SOP	Laboratory blank sample to assess potential for contamination from laboratory instruments or procedures.
	Laboratory Control Samples and Duplicates	Analyzed as per method requirements and laboratory SOPs	Evaluates laboratory reproducibility.

Table 7
Sample Container, Preservation and Holding Time Requirements

Matrix	Analytes	Bottles	Preservatives	Holding Time
Sediment	Metals, mercury	1 x 4 oz. plastic jar	Unpreserved	28 days
	Metals, other	1 x 4 oz. plastic jar	Unpreserved	6 months
	Sieve	1 x 16 oz. plastic jar	Unpreserved	N/A
	Hydrometer	1 x 16 oz. plastic jar	Unpreserved	N/A
	Atterberg Limits	1 x 16 oz. plastic jar	Unpreserved	N/A
	Hydraulic Conductivity	1 x gallon freezer bag	Unpreserved	N/A
	% Moisture	1 x 4 oz. plastic jar	Unpreserved	7 days
Interstitial Water	Metals, mercury	1 x 250 ml plastic jar	Nitric Acid ($HN0_3$)	28 days
	Metals, other	1 x 250 ml plastic jar	Nitric Acid ($HN0_3$)	6 months
	Cyanide	1 x 250 ml plastic jar	Sodium Hydroxide (NaOH)	14 days
	Oil & grease	1 x 1000 ml glass jar	Hydrochloric Acid (HCl)	28 days
	Phenols	1 x 1000 ml glass jar	Sulfuric Acid (H_2SO_4)	28 days
	pH	1 x 250 ml plastic jar	Unpreserved	Immediate
	BOD	1 x 1000 ml plastic jar	Unpreserved	48 hours
	TSS	1 x 1000 ml plastic jar	Unpreserved	7 days
	Phosphorus	1 x 250 ml plastic jar	Sulfuric Acid (H_2SO_4)	28 days