CONSTRUCTION QUALITY ASSURANCE PLAN

GRASSE RIVER, MASSENA, NEW YORK

Prepared for

Arconic, Inc.

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LIST OF ACRONYMS AND ABBREVIATIONS

µg/L	microgram per liter
Alcoa	Alcoa, Inc.
Arconic	Arconic, Inc.
ASTM	ASTM International
CCU	Cap Certification Unit
CQAP	Construction Quality Assurance Plan
CQC	Construction Quality Control
cy	cubic yard
dBA	A-weighted decibel
DER	Division of Environmental Remediation
DER-10	Technical Guidance for Site Investigation and Remediation
DMU	Dredge Management Unit
EDD	electronic data deliverable
FDR	Final Design Report
GAC	granular activated carbon
mg/kg	milligram per kilogram
mg/L	milligrams per liter
NFG	National Functional Guidelines
NTU	Nephelometric Turbidity Unit
NYSDEC	New York State Department of Environmental Conservation
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PM_{10}	particulate matter less than 10 microns
Power Canal	Massena Power Canal
QA	quality assurance
QC	quality control
QL	quantitation limit

RA	remedial action
RAO	Remedial Action Objective
RD	Remedial Design
ROD	Record of Decision
RSI	River and Sediment Investigation
RTK	real-time kinematic
SDG	sample delivery group
sf	square foot
SLF	Secure Landfill
SLWA	segment length-weighted average
SOP	standard operating procedure
SPDES	State Pollutant Discharge Elimination System
SRMT	Saint Regis Mohawk Tribe
Study Area or site	Grasse River Study Area
SVOC	semivolatile organic compound
T	Transect (e.g., T21)
TAT	turnaround time
TCL	target compound list
TSS	total suspended solids
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound

1 INTRODUCTION

In April 2013, the United States Environmental Protection Agency (USEPA) issued the Grasse River Superfund Site *Record of Decision* (ROD; USEPA, April 2013) that set forth the remedy USEPA selected to address polychlorinated biphenyls (PCBs) in the Grasse River Study Area (Study Area or site) in Massena, New York. In July 2013, Alcoa, Inc. (Alcoa; now Arconic, Inc. [Arconic]), submitted the Remedial Design (RD) Plan (Alcoa, July 2013), which provided the framework for developing the design necessary to implement the USEPA-selected remedy. The RD was completed in three stages documented in three consecutive reports: 1) the Preliminary Design Report (Alcoa, June 2015); 2) the Intermediate Design Report; and 3) the Final Design Report (FDR; this submittal).

This Construction Quality Assurance Plan (CQAP) is part of the FDR and establishes quality assurance (QA) and quality control (QC) procedures for the remedial work described in the FDR. It addresses remediation activities, including dredging, backfilling, capping, and post-dredge and post-cap verification sampling. The overall intent of the CQAP is to present approaches for planning, implementing, assessing, reporting, and assuring the quality of the project. If additional project elements are defined in the future or if refinements to this CQAP are necessary, a revised CQAP or CQAP Addenda may be submitted.

This CQAP establishes the requirements for the observations, testing, and documentation needed to verify that the work being performed (remedial and associated activities) meets or exceeds the standards established by the ROD, the RD, and project specifications. It contains descriptions of the routine and systematic inspections and tests to be conducted as part of the compliance assessment. It also presents procedures for the preparation, review, and transmittal of submittals controlling design activities, reporting and tracking deficiencies, and corrective actions.

1.1 Background

The Study Area is along the northern boundary of New York State in the Town and Village of Massena and encompasses approximately 8.5 miles of the Grasse River. It includes the following two components: 1) the 7.2-mile stretch of the river between the Massena Power Canal (Power Canal) and the St. Lawrence River, which is termed the lower Grasse River;

and 2) a 1.3-mile upstream background reach from just downstream of the Route 37 Bridge in Massena to the Power Canal confluence (Figure C1-1). For the purposes of the site investigations, 72 transects across the river have been delineated, beginning with Transect (T)1 near the upstream boundary and ending with T72 at the downstream boundary near the mouth of the river (Figure C1-2).

Detailed discussions of the Study Area's hydrology and hydraulics, floodplains, wetlands, river use, and fish habitat are provided in the *Comprehensive Characterization of the Lower Grasse River* report (Alcoa, April 2001) and the *Draft Addendum to the Comprehensive Characterization of the Lower Grasse River* report (Alcoa, April 2009), and a summary is included in Section 2 of the FDR.

1.2 Summary of Grasse River Remedial Design

As stated in the ROD (USEPA, April 2013), the major remedial action (RA) components of the USEPA-selected remedy are as follows:

- Dredging near shore sediment between T1 and T21 with sediment PCB concentrations greater than or equal to 1 milligram per kilogram (mg/kg) on a segment length-weighted average (SLWA) or maximum surface (0- to 12-inch) concentration basis, followed by backfill to grade
- Dredging near shore sediment between T21 and T72 with maximum surface (0- to 12-inch) sediment PCB concentrations greater than or equal to 1 mg/kg, followed by backfill to grade
- Placing an armored cap over the main channel sediments between T1 and T21 where either the SLWA or the maximum surface (0- to 6-inch) sediment PCB concentration is greater than or equal to 1 mg/kg
- Placing a main channel cap over sediments between T21 and T72 with maximum surface (0- to 6-inch) sediment PCB concentrations greater than or equal to 1 mg/kg
- Treating process water from the sediment dewatering facilities to meet New York State Department of Environmental Conservation (NYSDEC) discharge limits
- Monitoring (during remedial construction and long term) to demonstrate the effectiveness of the RA in meeting the Remedial Action Objectives (RAOs) outlined in the ROD, including monitoring of the Power Canal

- Air monitoring to ensure remedy implementation is protective
- Reconstructing habitat

2 PURPOSE AND SCOPE

2.1 Purpose

The purpose of this CQAP is to present a systematic management approach for the planning, implementation, control, and assessment of work to ensure that the end results satisfy the technical, regulatory, and quality objectives for the completion of the RA at the site. It was developed using the guidelines of *USEPA Requirements for Quality Assurance Project Plans* (USEPA, March 2001) and includes the policies, procedures, authorities, and requirements necessary for implementation and assessment of the RA and associated work.

This CQAP establishes a framework and criteria for establishing task-specific QC requirements and applies to work done by subcontractors, fabricators, and material or equipment suppliers. It also provides project-specific QA plans. Effective implementation of the QA and QC requirements, procedures, and reports within this plan will verify that the completed RA and associated work are in compliance with the provisions of project plans and specifications.

Requirements for the following are established in this document:

- QA and QC organization
- Personnel qualifications and training
- Procedures, guidelines, checklists, and other forms for conducting and controlling the RA and associated work
- Definable features of the work
- Reporting, record keeping, and documentation
- Inspections and tests
- Audits and surveillance
- Protocols for sampling and testing for monitoring the RA
- Deficiencies, noncompliance, and corrective actions

2.2 Scope

The RA proposed for the Grasse River consists of work performed in the fall of 2018 (Staged Cap Placement Test) and at the start of the 2019 construction season and continuing through the 2022 construction season. This CQAP has been developed for the RA, including related response activities such as operations, maintenance, and long-term monitoring.

Remedial work and associated activities described in this document include the following:

- Mobilization of dredging and capping equipment
- Removal (dredging) of near shore sediment
- Engineered capping in the main channel
  - Armored capping (T1 to T19)
  - Modified armored capping (T19 to T21)
  - Non-armored capping (T21 to T72)
- Placement of post-dredge backfill in the near shore
- Pre- and post-dredge surveying
- Pre- and post-cap surveying
- Sediment dewatering, stabilization and solidification, transport, and disposal
- Water treatment and discharge into the Grasse River
- Post-dredge verification sampling to verify that cleanup levels have been achieved
- Verification of cap and backfill material quality
- Post-cap verification sampling and testing to verify thickness and extent of engineered caps
- Post-backfill surveying and testing to verify elevation

All subcontractors and materials suppliers must comply with the sections of this CQAP specific to the work they are performing.

QA/QC associated with post-remediation site restoration will be covered in the restoration and reconstruction design documents. Additionally, QA/QC activities associated with Secure Landfill (SLF) operations are covered in the SLF design documents, and QA/QC activities associated with staging area development are covered in staging area design documents submitted under separate cover.

3 PROJECT MANAGEMENT

3.1 QA/QC Organization

This section describes and documents the organization and authority for the development, approval, and implementation of this CQAP, including verification of work activities. It also documents the organizational structure, functions, responsibilities, qualifications, levels of authority, and lines of communication that will be implemented as part of overall project QA.

3.1.1 General

QA is a management function used to verify that QC is properly performed and that the project objectives are met. QA will include review of QC documentation and testing on a spot or periodic basis. The primary goal of QA is to obtain a completed construction that meets the RAOs of the project.

QC involves performing the necessary tests, observations, and documentation to verify that the work being performed meets or exceeds the minimum standards established in the project specifications. QC includes the routine and systematic tests and inspections that are conducted to verify that each phase of the remedial work is in compliance with the applicable specification.

Project personnel are essential in achieving project success and will be responsible for identifying issues that could adversely affect the quality of the work. Project staff will be trained to understand the project requirements and be responsible for identifying, reporting, documenting, and verifying that the appropriate corrective actions have been implemented to ensure items and services conform to the applicable project specifications.

3.1.2 Organization, Duties, Responsibilities, and Authorities of QA/QC Personnel

The preliminary organization structure, showing the reporting and communication lines for each personnel category, is depicted in Figure C3-1. The majority of individuals who will fill these roles will be determined at a later date. It is the responsibility of the personnel involved in project activities that may affect the quality of data collection, design, operations,

implementation of RA, or other quality-related functions to be aware of and implement the quality policies and practices set forth by this CQAP.

USEPA. The USEPA will be the regulatory authority and agency responsible for overseeing and authorizing the RA described herein. In this capacity, the USEPA will review information described in the RD documents and this CQAP to ensure the project is implemented in a manner consistent with the approved project design documents. This may include review and approval of construction verification documentation, environmental monitoring data, and other QC information. The USEPA Remedial Project Manager (Young Chang) or her designee will exercise project oversight for the USEPA and coordinate with Arconic. The USEPA Remedial Project Manager shall identify any issues or problems if they should arise. Arconic will propose response measures or recommendations to the USEPA Remedial Project Manager or her designee as appropriate. The USEPA will make final decisions to verify compliance with the approved project design documents.

Agency Stakeholders. The USEPA will receive input from other project stakeholders, including the NYSDEC and the Saint Regis Mohawk Tribe (SRMT).

Arconic Program Manager (Larry McShea). The Arconic Program Manager will be the point of contact between the USEPA and the Arconic project team. Any issues related to QA or QC identified by the USEPA or the Arconic project team will be communicated to the Program Manager (or his designee), and he will work with the project team and the USEPA to resolve the issue in a timely manner.

Construction Project Manager (Mike Elsner). The Construction Project Manager will be the single point of contact for coordination of the project during construction. The Construction Project Manager will technically and administratively manage the project, and he will have authority over and be responsible for the following activities related to the construction QA program:

- Establishing and managing the QA organization
- Monitoring and controlling through oversight of project activities to ensure portions of the CQAP are implemented
- Maintaining an awareness of planning and scheduling QA/QC processes

- Establishing a system for managing records and documents for the project that ensures clarity, completeness, retrievability, conformance to the specifications, and document security

Construction Manager (Dan Casey). The Construction Manager will be responsible for the overall management of construction activities. The duties, responsibilities, and authorities of the Construction Manager will include the following:

- Issue stop-work order when necessary
- Monitor and control through surveillance of project activities to confirm that relevant portions of the plan are implemented
- Maintain an awareness of planning and scheduling of QA/QC processes
- Establish an overall records-management system for the project that provides clarity, completeness, retrievability, conformance to contract requirements, and document security
- Implement the approved site-specific plans
- Evaluate site-specific procedures and plans
- Evaluate the project schedule and budget

Project Field Engineer/Construction QA Officers (Kevyn Bollinger [River Operations] and Matt Millias [Upland Operations]). Based on the range of activities on the site, there will be separate Project Field Engineers/Construction QA Officers for river and upland activities. The Project Field Engineer/Construction QA Officers will provide or coordinate resources so that appropriate technical support is available during project implementation. They will also be responsible for the overall management of on-site QA and QC processes and procedures. The Project Field Engineers/Construction QA Officers will have the authority to act on quality matters and will be responsible for the maintenance and implementation of the CQAP. They will also have the authority to provide recommendations for changes to the design for approval by the Construction Project Manager. The duties, responsibilities, and authorities of the Project Field Engineers/Construction QA Officers will include the following:

- Coordinating appropriate reviews of the engineered plans and specifications prior to implementation

- Coordinating appropriate reviews and approvals for all Requests for Information that clarify the intent of the design
- Providing and maintaining an effective QA/QC system in accordance with this CQAP
- Reviewing and managing submittals in compliance with the project specifications and this CQAP
- Performing QA inspections to verify QC is being performed in accordance with this CQAP
- Preparing regular QA/QC Reports for project activities and submitting them to the Construction Project Manager
- Conducting or overseeing completion inspections of completed work
- Identifying items that do not conform to project plans and specifications and ensuring these items are completed
- Ensuring logs, records, standard procedures, and plans are distributed to the appropriate personnel
- Ensuring the document control system is maintained in a retrievable fashion and copies of applicable documents are transmitted to the appropriate personnel
- Verifying implementation of corrective activities and closeout corrective action documentation upon completion of corrective activities

QA Inspectors. The QA Inspectors will perform the construction and operation QA functions specified as task-specific QA requirements and will report directly to the Project Field Engineer/Construction QA Officer. They will observe the Remedial Construction Contractor as they perform the tasks, observations, testing, and documentation necessary to verify that project work meets or exceeds the minimum standards established by the project specifications. The type and number of QA personnel will be finalized during the construction planning phase. The duties, responsibilities, and authorities of the QA Inspectors may include the following:

- Assisting or representing the Project Field Engineer/Construction QA Officer in the performance of their duties when so directed
- Authorizing the issuance of Non-Conformance or Deficiency Reports and providing stop-work recommendations to the Project Field Engineer/Construction QA Officer
- Maintaining the daily log of inspections and providing the information necessary to complete the QA/QC Reports

- Inspecting and documenting the implementation of corrective actions
- Monitoring sampling and laboratory testing activities
- Performing field inspections of construction and operation activities

Surveyors and Samplers. Surveyors and Samplers will verify quality by conducting bathymetric surveys and collecting samples in accordance with this CQAP. They will be under the direction of the Project Field Engineer/Construction QA Officer.

Site Safety Representative (Toby Topa). The Site Safety Representative will be responsible for developing and implementing site-specific health and safety plans, tracking safety, and serving as the point of contact for employees and contractors for issues related to health and safety.

Remedial Construction Contractor (JF Brennan). JF Brennan has been selected as the Remedial Construction Contractor and will ensure the quality of the remedial construction and ensure construction meets the requirements of the plans and specifications.

Construction Engineers. The Construction Engineers will be responsible for coordinating with the Remedial Construction Contractor on specific remedial construction activities, including dredging, backfilling, capping, water treatment, and disposal of dredged material. Chuck Guest will serve as the lead Construction Engineer, providing home office support to the project and coordinating with other resources, as necessary.

QA/QC personnel will be trained and qualified in accordance with this CQAP and other project-specific requirements.

3.2 Procedures for Scheduling Inspections

3.2.1 General

These procedures are expected to establish controls and schedules for inspections and requirements for performing task-specific QC activities for definable features of work. A definable feature of work is a task that is unique and has separate control requirements.

Implementation of the three phases of control (preparatory, initial, and follow-up) allows for the Construction Project Manager and Project Field Engineer/Construction QA Officer to plan and schedule work to verify that the required actions have been completed, that the work is understood by the necessary personnel, and that the required materials and equipment are available. The three-phase inspection program will be implemented for the definable features of work expected to be completed during the project construction. The following activities are expected to require the three phases of inspection:

- Sediment sampling
- Water quality monitoring
- Air quality monitoring
- Noise monitoring
- Material source monitoring
- Sediment removal, processing, and disposal
- Water treatment
- Placement of backfill
- Placement of the main channel caps

The types of inspections performed for these tasks are described in the following sections. If other tasks are identified, procedures contained within task-specific plans for QC will be submitted as addenda or revisions to this CQAP as they are developed during the design process.

3.2.2 Preparatory Phase Inspection

Preparatory Phase Inspections will be performed prior to starting any definable feature of work. A Preparatory Phase Inspection meeting will be conducted by the Project Field Engineer/Construction QA Officer or designee and attended by the responsible construction personnel, including applicable QA/QC staff. The Preparatory Phase Inspection meeting will include the following:

- Reviewing material and equipment documentation for the required tests, submittals, and approvals
- Reviewing required control inspections and test requirements
- Confirming that the work to be tested or inspected is complete and conforms to the approved construction plans and specifications

- Confirming that the required materials and equipment for the inspections and tests are on hand and available for use, have been properly stored and calibrated, and are in proper working condition
- Establishing acceptable levels of workmanship needed to meet the requirements of the plans and specifications

Preparatory Phase Inspection meetings will be documented in meeting minutes prepared by the Project Field Engineer/Construction QA Officer. These meetings will be noted in the QA/QC Reports.

3.2.3 Initial Phase Inspection

An Initial Phase Inspection will be conducted by the Project Field Engineer/Construction QA Officer or designee at the beginning of each definable feature of work (e.g., near shore dredging, backfilling, and main channel capping). The inspection will be performed as soon as it is determined that a sufficient portion of the definable feature of work has been accomplished to evaluate the following criteria:

- Compliance with the project specifications, engineered plan drawings, and submittals
- Acceptable levels of workmanship
- Compliance with material specifications
- Resolution of differences

The Initial Phase Inspections will include the participation of the responsible personnel, including the subcontractor's QC personnel involved with the definable feature of work. Initial Phase Inspection meetings will be documented on meeting minutes prepared by the Project Field Engineer/Construction QA Officer using an Initial Phase Inspection Meeting checklist. These meetings will be noted on the QA/QC Reports.

3.2.4 Follow-up Phase Inspections

Follow-up Phase Inspections will be performed as deemed necessary. Any deficiencies will be corrected as soon as possible prior to starting the new work, or they will be included on deficiency punch lists. Follow-up Phase Inspection Reports will be documented on the QA/QC Reports by project QC personnel.

3.2.5 Completion Inspection

At the completion of the definable feature of the work, the feature will be inspected for compliance with the plans and specifications. The Project Field Engineer/Construction QA Officer is responsible for initiating the completion inspection and for verifying the development of a deficiency punch list, when necessary, to identify any non-conforming or incomplete work. Upon completion of the punch list, a second inspection will be conducted to verify that the items conform to requirements. Documentation of the completed punch list items will be provided to the Project Field Engineer/Construction QA Officer and included in the QA/QC Reports.

3.2.6 Inspection Documentation

The Project Field Engineer/Construction QA Officer will be responsible for maintaining the inspection records. Inspection records will provide the information necessary to clearly verify that the items or activities inspected conform to the specified requirements or, in the case of non-conforming conditions, provide evidence that the conditions were brought into conformance. During construction, inspection documentation will be stored on site. QA/QC documentation will be archived annually.

3.3 Procedures for Scheduling, Reviewing, and Managing Submittals

3.3.1 Responsibilities

The Construction Project Manager will be responsible for the preparation and maintenance of the required project submittals. Submittals will be listed in the project Submittal Register, which will be updated weekly by the Remedial Construction Contractor and distributed monthly by the Project Field Engineer/Construction QA Officer.

3.3.2 Scheduling of Submittals

The Construction Project Manager, in conjunction with the Project Field Engineer/Construction QA Officer, will review the project plans and specifications for required submittals and direct the preparation of the Submittal Register to ensure that submittals are made.

3.3.3 Review of Submittals

The Construction Project Manager and Project Field Engineer/Construction QA Officer, or authorized designees, will review submittals for completeness and accuracy. Any submittals requiring modifications or changes will be returned to the originating organization for correction, then resubmitted for review and approval by the Project Field Engineer/Construction QA Officer and Construction Project Manager prior to submittal to the Agencies as appropriate.

3.3.4 Transmittal

Submittals will be sent under the cover of a transmittal form or cover letter in the quantities required. The transmittal form or cover letter will reference the appropriate specification sections or reference documents.

3.3.5 Document Management System

A document control system will be developed and maintained. The document control system will include measures to file and store appropriate project information, including all types of correspondence (conventional mail, fax, and email), project plans, submittals, and other records. Project documents will be dated and numbered and identify the current revision status as appropriate. Obsolete documents will be marked as such or removed from files to prevent unintended or inappropriate use.

3.3.6 Requirements for Suppliers of Materials and Services

This CQAP establishes a management system to verify that procured items and services conform to the requirements identified in the plans and specifications. The system applies to items and services that have an impact on the quality of the requirements specified in the project design documents. The basic management system includes the following elements (as appropriate) for the specific item or service purchased:

- Identification of quality requirements for services, materials, and equipment
- Source evaluation and selection

- A method for confirming the level of supplier quality through inspections, tests, certifications, source inspections, and other valid means of verifying compliance of items and services
- Documentation and implementation of quality programs
- Requirements for suppliers to incorporate appropriate quality requirements into their sub-tier procurement documents
- Provisions for assessing, monitoring, and controlling the performance of suppliers through periodic audits, source inspections, or receipt inspection upon delivery (as appropriate)
- For materials and equipment inspections, verify proper configuration; identification; dimensional, physical, and other specified characteristics; freedom from damage; and cleanliness using specified quantitative or qualitative accept/reject criteria
- If deficiencies are identified, the inspector shall perform applicable reinspection of the item (i.e., supplies, equipment, and services) or activity after the supplier completes any required reinspection and records the inspection test results

3.3.7 Contractor Submittals

The Project Field Engineer/Construction QA Officer will be responsible for the implementation of inspections, surveillance, document reviews, and audits for monitoring the Remedial Construction Contractor's QC activities and associated submittals to verify compliance with the project plans and specifications. These activities will be documented on inspection reports, pre-established checklists, audit reports, field logs, or other forms appropriate to the function performed.

The Remedial Construction Contractor's QC staff will provide QC checks during the three phases of control to determine compliance with the QC requirements set forth in applicable documents, including the following:

- Meeting quality requirements
- Generating, controlling, and maintaining required documentation
- Performing and documenting required inspections and tests
- Identifying, reporting, and correcting non-conformance conditions

4 DATA GENERATION AND ACQUISITION

4.1 Materials Management

A receiving inspection is one method used to accept an item from a supplier. Receiving inspection requirements developed from the procurement documents will be compared with all documents provided with the shipment to verify that materials received comply with procurement documents. Other methods of acceptance are source verification, supplier certificate of conformance, post-installation tests, or a combination of any of the former. Quality receiving inspection requirements that state accept/reject criteria must be established in accordance with the procurement documents.

4.2 Control Testing Procedures

One of the primary CQAP elements is the design of a post-construction verification plan for assessing compliance with the project plans and specifications to achieve the RAOs defined by the ROD. This includes a post-dredge verification sampling program to confirm the attainment of the specified design elevations for near shore dredging, post-backfill verification measurements of the post-dredge backfill elevation, and post-cap verification measurements for minimum thickness and coverage for main channel capping. Specific responses to be considered in the event of non-conformance with the design requirements will be identified in the project plans.

This section presents a summary of plans developed to verify the performance of RAs within the Grasse River relative to the plans and specifications in accordance with the ROD requirements. The verification plans and performance criteria presented in the following sections build on similar plans and criteria that have been used in other large-scale sediment projects and address the following objectives:

- Near shore dredging
  - Verification that dredging has been completed to the horizontal and vertical extents required by the design
  - Verification that dredging has addressed the 1 mg/kg cleanup criteria for PCBs with appropriate measures for post-dredge residuals

- Post-dredge backfilling
  - Verification that post-dredge backfilling has been completed to the horizontal extents and within the allowable tolerances around pre-dredging grade
- Main channel capping
  - Verification that capping has been completed to the horizontal extents and minimum thicknesses and within the allowable tolerances as required by the design

Post-dredge verification sampling is discussed in detail in Section 4.3, post-dredge backfilling verification sampling is discussed in detail in Section 4.4, and capping verification sampling is discussed in detail in Section 4.5.

4.2.1 Testing Procedures

Testing is an important part of the quality program. QC testing will be performed to determine whether procedures and processes are producing the desired results. The task-specific QC requirements will list the project or task-specific required QC tests and specify whether the tests are to be performed by an independent approved testing laboratory or by personnel on site. The scope of the QC testing will also be addressed, and personnel involved with the specific task will be aware of the methods to be used and the extent of the testing. In addition, test reporting requirements will be defined, and facilities and testing equipment will be verified, calibrated, and accepted. Assigned personnel will be qualified as appropriate for specific tasks. Testing procedures will be developed, approved, and implemented to perform the tests specified for each of the project's definable features of work as applicable or specified. The type, number, and frequency of the tests will be as specified and will include the requirements of referenced standards or regulatory guidelines.

4.2.2 Testing Documentation

Testing performed during project activities will be documented on QC test forms, which will be developed from task-specific QC requirements and include the following information:

- Test name, procedure, and frequency
- Specification reference
- Responsible laboratory and personnel

- Acceptance criteria
- Testing procedures
- Calibration methods and frequency
- Test documentation and results

Testing activities and results of the tests and monitoring activities will be included in the QA/QC Report. Test reports, calibration records, and other recording forms used to document the test activities will be maintained in the project files.

4.3 Dredging

The verification plans and performance criteria for dredging address the following objectives:

- Verification that dredging has achieved the horizontal and vertical extent required by the design
- Verification that dredging has addressed sediments exceeding 1 mg/kg PCBs in designated near shore dredge areas with appropriate measures for post-dredge residuals

4.3.1 Removal Verification

This section discusses how post-dredge verification sampling data will be collected in near shore dredging areas and how these data will be evaluated to determine alternatives for management of dredging residuals. All post-dredge verification sampling will be performed in accordance with applicable standard operating procedures (SOPs), which will be developed during future design phases.

4.3.1.1 Dredge Management Units

Near shore dredge areas have been subdivided into Dredge Management Units (DMUs) based on location and operational considerations in order to facilitate timely completion of activities within individual work units. The DMUs were developed in coordination between Anchor QEA and the remedial contractor considering anticipated production rates and the spatial conditions. In total, 56 DMUs were designated for the dredging operations. It is

anticipated that two to three DMUs will be closed on average each week during the dredging season. The following factors were considered in development of the DMUs:

- DMUs were separated by the north or south shoreline.
- DMUs were designed to not extend more than 3.5 transects along the river.
- Where not limited geographically, individual dredge areas were combined to form DMUs with a target design dredge volume of about 2,000 to 3,000 cubic yards (cy), which are estimated to require 5 to 8 days dredging based on an average estimated dredge production rate of 375 cy/day per dredge. Large, contiguous dredge areas were separated into multiple DMUs to achieve this target design volume. Discrete dredge areas necessitated smaller DMUs.
- Distinct DMU IDs were assigned for ease of tracking with a prefix to differentiate its location along the north or south shoreline (e.g., DMU-N01, DMU-S02).
- Each individual discrete area comprising a DMU was also assigned an alphabetical suffix for tracking purposes.

Attachment A includes figures showing the DMUs that will be used to verify completion of the dredging.

4.3.2 Removal Verification Approach

Post-dredge bathymetric surveys will be performed using a multibeam or single-beam acoustical system or manual survey method depending on water depth and vessel access limitations, in accordance with Specification Section 02 21 00 – Surveys (Appendix B of the FDR). The same survey method will be used for pre- and post-dredge surveys within a given area. For instance, if a pre-dredge multibeam survey is performed for a given area, then the post-dredge survey will also use multibeam methods. Unedited survey data will be processed by the Remedial Construction Contractor and Project Field Engineer/Construction QA Officer to verify attainment of target elevation and dredge volumes.

The first step of post-dredge verification is to establish whether the required design cutline has been met. The proposed approach to verifying compliance with the design is based on achieving the required grade across at least 95% of the DMU area, excluding locations where rock or clay (i.e., high subgrade) constrained the depth of dredging. If the cutline has not

been met per this compliance standard, then additional dredging will be performed. High subgrade survey procedures are described in Section 4.8.

The cutline evaluation will be conducted on 10-foot by 10-foot grid cells. The smallest practical area to assess the difference in thickness is a 10-foot by 10-foot area given that the uncertainty in comparing individual point measurements from multiple acoustical surveys is approximately plus or minus 6 inches. In areas where multibeam surveys cannot be conducted (i.e., where single-beam or manual survey methods are necessary), the spacing of the measurements will consider the local conditions on a location-by-location basis. The spacing may be reduced as appropriate to collect adequate data to characterize the conditions. For instance, in a DMU with a narrow width (10 to 15 feet), at least two survey measurements will be collected across the width of the DMU. In areas where manual survey methods are necessary, measurements will be collected along transects perpendicular to the shore and spaced at approximately 10-foot intervals, with measurements collected every 2 to 5 feet along the transect line.

The dredge verification evaluation will be conducted by comparing the average value of each 10-foot by 10-foot grid cell from the post-dredging survey dataset with the average value of the required elevation dataset (based on the Dredge Prism XYZ File) in each corresponding grid cell. Field-identified locations where rock or clay (i.e., high subgrade) constrain the depth of dredging, as accepted by the Construction Manager, will be considered to have achieved the required elevations. Survey data processing will be constrained to the horizontal extents of the remediation boundaries to avoid edge effects with averaging data inside and outside of the boundaries. Cells will be considered compliant with the design if the average post-dredge elevation is at or below the average design cutline elevation within the 10-foot by 10-foot grid cell.

If more than 5% of the cells within a DMU are non-compliant, additional dredging will be conducted in non-compliant cells to achieve the 95% requirement for the DMU. In addition, if the average elevation of a single 10-foot by 10-foot grid cell is more than 3 inches above the target elevation, that grid cell will be re-dredged everywhere except for areas where clay or hard bottom (i.e., high subgrade) have been encountered.

After surveying confirms that the target dredge cutline has been achieved for at least 95% of the DMU area, sediment core samples will be collected in designated areas, as described in Section 4.3.3, to characterize the post-dredge sediments and determine whether an additional dredging pass is required.

4.3.3 Post-Dredge Sampling

Following acceptance of the cutline criteria, sediment cores will be collected on a pre-defined grid in a given DMU. The required nominal sampling density will be eight cores per acre of DMU, with locations offset from existing design cores. A greater density of sampling will be employed, where necessary, so that each discrete dredge area within a DMU has at least one sample. Therefore, in cases where a DMU makes up multiple individual design areas, post-dredge sampling will include at least one sample in each area. Cores will be advanced to refusal and segmented in 6-inch intervals. The top 18 inches (i.e., three segments; depending on core recovery) will be analyzed for PCBs, and any remaining sections will be archived pending the results of the upper segments. Attachment B presents the planned locations for post-dredge sampling.

The approach for interpreting these results is outlined in the decision tree shown on Figure C4-1. Verification will be assessed, and potential follow-up actions determined, based on the average PCB concentration for each 6-inch interval relative to the 1 mg/kg cleanup criterion, as described in the following summary:

- If the post-Pass 1 DMU average PCB concentration exceeds 10 mg/kg at any depth, locations causing the average to exceed criteria will be re-dredged (second dredge pass), and the area will be re-surveyed and assessed as described in Section 4.3.2 to verify compliance with the target re-dredge elevation. Additional post-dredge sampling will be conducted to determine whether additional re-dredging is warranted. Within these areas, there is a possibility for a total of up to three dredge passes (i.e., one design pass and up to two re-dredge passes)[1].

[1] If an individual sample point with a high residual PCB concentration is identified after the first dredge pass in a DMU that achieves compliance with the Dredge Verification Decision Tree, the information will be reviewed with EPA and a decision made as to whether a second dredge pass is warranted.

- If the post-Pass 1 DMU average PCB concentration is between 5 and 10 mg/kg at the surface (top 6 inches) or between 1 and 10 mg/kg in the subsurface (below 6 inches), locations causing the average to exceed criteria will be re-dredged. After the second dredge pass, samples will be collected for documentation purposes only, and backfilling activities will commence. Within these areas, there is a possibility for a total of two dredge passes (i.e., one design pass and one re-dredge pass).
- If the post-Pass 1 DMU average PCB concentration is below 5 mg/kg at the surface and below 1 mg/kg in the subsurface, backfilling activities will commence. Within these areas, only one dredge pass (i.e., the design pass) would occur.

4.4 Backfill Verification

Post-dredge backfill will be installed following acceptance of the required dredging within a DMU. The following QA/QC steps will be performed relative to backfilling:

- Verification of Import Material Quality: The chemical and physical characteristics of the materials to be used for backfilling will be verified for their intended use (see Section 4.5.1 for details).
- Verification of Required Grade: The satisfactory placement of backfill material over the required areas and to the required grade to match pre-dredge conditions (with localized exceptions in steep slope areas) will be verified.

Bathymetric surveying and equipment positioning records will be used to verify proper placement of backfill where required by the design. It may be necessary for post-dredge backfill to extend laterally up to or beyond the estimated lateral boundary of the backfill areas. Achievement of lateral extent will be demonstrated through pre- and post-placement bathymetric surveys and GPS records from backfill construction.

For post-dredge backfilling, achievement of the required grade will be demonstrated by verifying that tolerances around pre-dredge grade are met for a specified DMU. Placement of the minimum required grade of post-dredge backfill will be verified primarily by bathymetric surveying. Maps will be developed following the placement of the backfill material comparing the final, placed surface to the pre-dredging surface. These maps will be

based on multibeam, single-beam, or manually collected survey data. This information will be provided to the Agencies as maps.

Backfill elevation compliance will be assessed on the same 10-foot by 10-foot grid cell layout used for dredge verification (again, constrained to the remediation boundaries). Survey data within each 10-foot by 10-foot grid cell will be averaged to produce a single elevation value for each cell. The average post-placement grid cell elevations will then be compared with the average pre-dredging survey elevations for each corresponding grid cell.

Additional backfill material will be placed if more than 10% of the grid cells in the DMU are below the pre-dredge grade (with an allowable tolerance of minus 3 inches), or if any 10-foot by 10-foot grid cell average elevation is more than 3 inches below the allowable backfill tolerance (i.e., more than 6 inches below the pre-dredge grade).

If backfill material is overplaced in more than 10% of the DMU above the pre-dredge grade (with an allowable tolerance plus 3 inches), or if any 10-foot by 10-foot grid cell average elevation is more than 3 inches above the allowable backfill tolerance (i.e., more than 6 inches above the pre-dredge grade), the Remedial Construction Contractor will be directed to remove material as necessary to achieve compliance within each DMU.

Special considerations will be made for backfill areas located along side-slope boundaries or in other areas with steep slopes. In these areas, verification will be evaluated on a case-by-case basis considering specific field conditions, which may include exemptions or adjustments to acceptance criteria for these areas).

In areas where amended backfill is used (see Section 5.4 of the FDR), a sample of the amended backfill will be collected prior to placement for off-site laboratory analysis of the granular activated carbon (GAC) content using the density separation test method (see Attachment C for testing SOPs).

4.4.1 Backfill Placement Test

At the start of backfill placement, the Remedial Construction Contractor will be required to demonstrate the efficacy of material blending and placement methods in the first DMU. Backfill placement within this DMU will be carefully tracked and monitored with a focus on placement methods, operational controls, and water quality. If necessary, the Remedial Construction Contractor will be required to make refinements to the material blending and/or placement process, and an additional backfill placement test will be required to demonstrate achievement of the required results. The Remedial Construction Contractor will also be required to repeat the backfill placement test if placement means and methods are changed during the course of the work or if requested by the Construction Manager.

4.5 Cap Verification

During remediation of the Grasse River, engineered caps will be installed in the main channel of the river. Placement of the armored cap in the T1 to T21 reach of the river (including a modified armored cap between T19 and T21) will require the use of various materials and placement technologies based on the multilayered engineered cap requirements. The following QA/QC steps will be performed relative to engineered caps:

- Verification of Import Material Quality: The chemical and physical characteristics of the cap materials will be verified for their intended use (Section 4.5.1).
- Verification of Specified Thickness and Extent: The satisfactory placement of the capping materials over the required areas and to the required thicknesses and extents provided by the design will be verified.

The following are the three reaches for cap placement:

- T1 to T19: Between T1 and T19, the design calls for an armored cap, comprising 6 inches of a sand-based chemical isolation layer that will be overlain by 6 inches of gravel filter layer and 13 inches of gravel and cobbles (3- to 10-inch-diameter particles), as well as a sand-based habitat layer in portions of the area.
- T19 to T21: Between T19 and T21, where soft, sensitive clay soils exist, the design calls for a modified armored cap comprising 6 inches of a sand-based chemical isolation layer overlain by 12 inches of gravel armoring.

- Downstream of T21, the main channel cap comprises 12 inches of a sand-based chemical and physical isolation and habitat layer with no additional armoring.

Separate approaches may be used for verification of caps upstream of T21 and those downstream of T21 based on differences in the cap materials.

Achievement of the required thickness and lateral extent will be demonstrated primarily through evaluations of pre- and post-placement bathymetric surveys and placement equipment positioning records. This information will be provided to the Agencies as maps in hardcopy and electronic formats. Alternative methods for thickness verification, including cores, catch pans, and grade stakes, may be used in lieu of, or to supplement, bathymetric surveys depending on site conditions and accuracy of the measurements.

4.5.1 Verification of Import Material Quality

Imported materials used for engineered caps and post-dredge backfill must meet the requirements described in the specifications. Prior to the use of any imported material, a Borrow Source Characterization Report will be prepared for each imported material to verify its specified physical properties, chemical properties, and gradation. Verification of the physical and chemical quality of the imported materials will be performed at the source location prior to shipment to the site using the following approach.

Cap Chemical Isolation Layer, Coarse Chemical Isolation Layer, Slope Grading Fill, Modified Armor Layer, Gravel Filter Layer, Topsoil, Post-Dredge Types A and B Backfill Material, and Wood Organic Material

- One representative sample from each distinct source and one sample per 20,000 tons delivered to the site will be submitted for the following analyses (additional samples will be submitted for these analyses if the source of a material changes):
  - Target compound list (TCL) volatile organic compounds (VOCs; USEPA SW-846 Method 8260C); samples for volatile analysis will be collected in accordance with USEPA SW-826 Method 5035
  - TCL semivolatile organic compounds (SVOCs; USEPA SW-846 Method 8270D)
  - Pesticides (USEPA SW-846 Method 8081B)

  - PCBs (USEPA SW-846 Method 8082A)
  - Herbicides (USEPA SW-846 Method 8151A)
  - Target analyte list metals (USEPA SW-846 Method 6010C/6020A/7471B)
  - Cyanide (USEPA SW-846 Method 9012B)
  - Total organic carbon (for chemical isolation and coarse chemical isolation layers only; USEPA SW-846 9060A Modified)

- One sample per 10,000 tons of Type A Backfill (sand and topsoil mixture) and blended Habitat Layer Material (sand and Wood Organic Material Mixture) will be submitted for organic content analysis (ASTM International [ASTM] D2974)
- In addition to the analyses described in this section, analyses of the following geotechnical tests will be performed on representative samples from each distinct source for every 10,000 tons delivered to the site, unless otherwise noted:
  - Grain size distribution (ASTM D6913 or ASTM C136)
  - In situ moisture content (ASTM D2216)
  - Weight per unit volume of uncompacted materials (ASTM C29 or equivalent); one sample per 35,000 tons
  - Particle-specific gravity (ASTM D854, ASTM C128, or ASTM C127 depending on particle size); one sample per 35,000 tons

Cap Armor Layer, Cap Filter Layer, and Type C Backfill Material

- A representative sample from each distinct source will be submitted for the following analyses for every 10,000 tons delivered to the site unless otherwise noted:
  - Grain size distribution (ASTM D6913, ASTM C136, or alternate approved by the Construction Manager)
  - Los Angeles abrasion and impact testing (ASTM C131)
  - Sodium Sulfate Test for Soundness (ASTM C88)
  - Weight per unit volume of uncompacted materials (ASTM C29, or equivalent)
  - pH (USEPA SW-846 9045D)

Additionally, individual delivery loads will be visually inspected for any objectionable content (e.g., concrete, brick, construction debris, or other deleterious material) to ensure compliance with the general physical requirements of the design.

4.5.2 Cap Certification Units

Main channel cap areas will be subdivided into Cap Certification Units (CCUs) based on location and operational considerations in order to track and facilitate timely completion of activities within individual work units. The CCU layouts will be developed in coordination with the selected Remedial Construction Contractor and used as a basis for verifying compliance with cap placement criteria. The CCUs will provide complete coverage of the required capping areas. They will generally be 1 to 5 acres in size and will be aligned to include only one cap type (e.g., armored cap, modified armored cap) per CCU. CCUs will be provided for agency review prior to 2020 capping operations.

4.5.3 Verification of Cap Thickness and Lateral Extent

4.5.3.1 Main Channel Armored Cap (T1 to T19)

Placement of the minimum specified thicknesses and extent of each engineered cap layer for the armored cap placed between T1 and T19 will be verified using a combination of physical measurements (core samples and catch pans) and bathymetric surveying. Selection of the most appropriate verification measurement technique will be determined prior to or during the initial stages of construction (e.g., during the startup demonstrations) and may be varied as construction proceeds depending on local site conditions. However, physical measurements (i.e., core samples and catch pans) are expected to be used as the primary verification method for the sand and gravel cap layers, and bathymetric surveys will be used to supplement the data as needed. Because physical thickness measures of cobble armor layer are not practical, bathymetric surveys will be used to verify the minimum thickness of the armor layer. Records of volumetric placement and equipment positioning will also be used as an additional line of evidence to verify proper placement of main channel armored caps to the extents required by the design.

In some portions of the capping areas, staged placement of the layers will be required due to the presence of existing low-strength clay material. The staged placement will include specified waiting periods between lifts of the cap in order to provide adequate time for consolidation and strength gain in the clay. For extended cap waiting periods, additional surveys or cap thickness measurements will be performed following the waiting period to confirm the cap layer has not eroded.

Post-placement thickness measurements of sand and gravel layers of the placed caps will be conducted using cores or catch pans distributed across a CCU at the following frequency:

- Phase 1: At the beginning of each construction season or when placement methods change, eight in situ measurements (i.e., cores or catch pans) will be collected per acre for thickness verification.
- Phase 2: If two successive sets of measurements collected during Phase 1 meet thickness compliance criteria, then the measurement frequency thereafter will be four in situ measurements per acre for thickness verification as long as consistent cap placement methods are used.

Physical measurement locations will be randomly selected, constrained only by the lateral extent of the CCU and the position relative to other measurement locations (i.e., to provide spatial distribution). When coring devices are used as the standard to measure applied cap chemical isolation layer thickness, the applied thickness will be determined from visual observation of the clean (unmixed) sand thickness plus 50% of the mixing zone thickness where the sand comprises at least half of the matrix, as determined through visual observations or physical characterization in the field. When using catch pans as the standard to measure the applied cap thickness for any material the applied thickness will be determined as the average of a minimum of 2 measurements across the catch pan.

Placement of an individual cap layer will be considered complete within a CCU if the minimum required design thickness has been achieved in at least 95% of the thickness measurements in that CCU, and any remaining measurements are 90% or greater of the minimum design thickness. For example, for a CCU that is 2.5 acres where 20 thickness measurements are collected within the CCU and the minimum required cap layer thickness is 6 inches, at least 19 of those measurements must be at least 6 inches of cap material, and the remaining measurement must be at least 5.4 inches of cap material.

In addition to the physical measurement of layer thickness, bathymetric surveys will be performed after placement of each cap layer. For the sand chemical isolation and gravel filter layers of the armored cap between T1 and T19, the bathymetric surveys will be used to verify uniform material placement and thereby supplement the physical measurements (using cores or catch pans) for compliance. However, bathymetric surveys will not be used

as a measure of cap thickness for assessing compliance with the design. If any non-conforming areas are identified, additional physical measurements will be conducted to assess the layer thickness, and additional material will be placed if deemed necessary.

Because physical thickness measurements will not be practical for the cobble armor layer upstream of T19, the bathymetric surveys will serve as the primary compliance measurement for armor layer thickness.

Similar to verification of the backfill placement, bathymetric surveys following cap placement will be processed on a 10-foot by 10-foot grid. Survey data within each 10-foot by 10-foot grid cell (or portion thereof when constrained by the remedial boundaries) will be averaged to produce a single average elevation value for each cell. Cap thickness difference (isopach) maps will be developed following the placement of each of the armored cap's three lower layers (6 inches of a sand-based chemical isolation layer, 6 inches of gravel filter layer, and 13 inches of gravel or cobble armor stone), but not after the surficial habitat layer (see the remainder of this section). The difference maps will be based on pre- and post-placement multibeam survey data within each grid cell. These maps will also be used to compare the extents of placement to the required design limits.

For the sand and gravel layers, cores will be used to verify cap thickness as discussed previously. The isopach maps will be reviewed to identify any evidence of potential uneven layer placement and to compare the extents of placement to the required design limits. If uneven placement is identified or if the design extents have not been achieved, additional physical measurements will be conducted in these areas to assess the layer thickness, and additional material will be placed if deemed necessary. If additional cap material is added, measurements will be taken to verify that the total placed cap thickness is compliant with the design.

For the armor layer, additional material will be placed if more than 5% of the grid cells in the CCU do not meet the required thickness based on the isopach maps, or if any 10-foot by 10-foot grid cell is more than 6 inches below the required thickness.

In addition to the cap thickness measurements for verifying compliance described in this subsection, the remedial contractor will also perform QC checks throughout construction.

These QC checks are expected to include continual tracking of material volumes placed in a given area, as well as a high frequency of physical samples (i.e., approximately 100 cores or up to 60 catch pans per acre) to provide feedback on equipment operations. These QC field measurements will be documented in logbooks, field forms, or an electronic database and will be used as an additional line of evidence for evaluating compliance with the minimum required design thickness. Specifically, if any of the physical thickness measurements for compliance indicate less than the minimum required layer thickness, the QC data will be reviewed to corroborate the compliance data and determine appropriate response actions.

A habitat layer will be placed on top of the armor stone over the armored cap area between T6 and T19. The habitat layer will be designed to fill the interstitial voids between the armor stone and leave 1 to 2 inches of material on top of the stone. Placement of the habitat layer will be verified by measuring the volume of material placed per unit area, as documented in Remedial Construction Contractor placement records in accordance with Specification Section 31 23 23 – Capping and Backfilling (Appendix B of the FDR).

4.5.3.2 Main Channel Modified Armored Cap (T19 to T21)

Placement of the minimum specified thicknesses and extent of each engineered cap layer for the modified armored cap placed between T19 and T21 will be verified primarily through physical measurements (cores and catch pans) and supplemented by bathymetric surveys, similar to the approach described in Section 4.5.3.1 for the sand and gravel layers of the armored cap between T1 and T19. Records of volumetric placement and equipment positioning will also be used to verify proper placement of main channel armored caps to the extents required by the design. As discussed in Section 4.5.3.1, additional surveys or cap thickness measurements may be performed in areas where staged cap placement is necessary.

Post-placement thickness measurements of sand and gravel armor layers of the caps placed between T19 and T21 will be conducted using cores or catch pans distributed across a CCU at the frequencies outlined in Section 4.5.3.1. Placement of an individual cap layer will be considered complete within a CCU if the minimum required design thickness has been achieved in at least 95% of the thickness measurements in that CCU and if any remaining measurements are 90% or greater of the minimum design thickness.

Bathymetric surveys will also be performed after placement of each layer, and difference maps will be created based on pre- and post-placement average grid cell elevations for each 10-foot by 10-foot grid cell (or portion thereof when constrained by the remedial boundaries). Cap thickness difference maps will be developed following the placement of each of the modified armored cap's two lower layers (6 inches of a sand-based chemical isolation layer and 12 inches of armor layer). These difference maps will be reviewed to verify uniform material placement across the CCU, but will not be used as a measure of cap thickness for assessing compliance with the design. If any non-conforming areas are identified, additional physical measurements will be conducted to assess the layer thickness, and additional material will be placed if deemed necessary. These maps will also be used to compare the extents of placement to the required design limits.

Consistent with the approach for the armored cap between T1 and T19, the contractor will also perform QC checks throughout construction that will be used as another line of evidence to evaluate and verify cap thickness.

4.5.3.3 Main Channel Cap (T21-T72)

Placement of the minimum specified thicknesses and extent for each of the two layers of the main channel cap placed downstream of T21 will be verified primarily by physical measurements and supplemented by bathymetric surveying, similar to the approach described in Section 4.5.3.1 for the sand and gravel layers of the armored cap between T1 and T19. Records of volumetric placement and equipment positioning will also be used to verify proper placement of main channel caps to the extents required by the design.

Post-placement thickness measurements of the caps placed between T21 and T72, which include 6 inches of a sand-based chemical isolation layer and 6 inches of a sand-based physical isolation/habitat restoration layer, will be conducted using cores or catch pans distributed across a CCU at the frequencies outlined in Section 4.5.3.1.

Placement of the sand chemical isolation layer will be considered complete within a CCU if the minimum required design thickness has been achieved in at least 95% of the thickness

measurements in that CCU and if any remaining measurements are 90% or greater of the minimum design thickness.

The placement of the habitat layer above the chemical isolation layer will be assessed using physical measurements (i.e., cores and catch pans), but the compliance criteria will be based on achievement of the total main channel cap thickness of 12 inches (including the placed and verified chemical isolation layer) rather than the independent thickness of the habitat layer. To verify the minimum 12-inch thickness, cores will be advanced through the entire cap thickness (both chemical isolation and habitat layer) at the same frequency as described in Section 4.5.3.1. This approach will confirm that the minimum required thickness for chemical isolation has been placed consistent with the design, confirm that the minimum total cap thickness of 12 inches has been achieved.

Bathymetric surveys will also be performed after placement of each layer and difference maps will be created based on pre- and post-placement multibeam survey data averaged for each 10-foot by 10-foot grid cell. These difference maps will be reviewed to verify uniform material placement across the CCU but will not be used as a measure of cap thickness for assessing compliance with the design. If any non-conforming areas are identified, additional physical measurements will be conducted in these areas to assess layer thickness, and additional material will be placed if deemed necessary. These maps will also be used to compare the extents of placement to the required design limits for each cap lift. Consistent with the approach for the armored cap between T1 and T19, the remedial contractor will also perform QC checks throughout construction, which will be used as another line of evidence to evaluate and verify cap thickness.

4.5.3.4 Main Channel Caps in Areas of Irregular Bottom (T1-T72)

In localized areas, the existing river bottom is uneven or has steep slopes that may limit the ability to place a uniform cap layer. For instance, some portions of the main channel capping areas contain outcrops of till and boulders in the form of steep-sided ridges or other formations. Placement of a uniform thickness cap over these areas is not feasible or even necessary given the nature of the material (e.g., rock versus sediment). Therefore, the design requires placement of double the thickness of the chemical isolation layer (minimum 12-inch

instead of the minimum 6-inch thickness) within these localized areas of the main channel where an irregular outcrop ridge is prevalent. Placement of the minimum cap thickness in these areas specifically identified on the engineering plan drawings will be verified on a volumetric basis through review of Remedial Construction Contractor placement records demonstrating that the specified volume of material has been placed over a unit area.

4.5.4 Cap Placement Test

At the start of cap placement each construction season, the Remedial Construction Contractor will be required to demonstrate the efficacy of material blending and placement methods. The Remedial Construction Contractor will first demonstrate through on-site "bench-scale" testing that cap materials (including amendments) have been adequately prepared (including pre-soaking) and will uniformly settle through a column of water without visible segregation to the satisfaction of the Construction Manager. Following approval of the upland testing by the Construction Manager, the Remedial Construction Contractor will demonstrate placement in the first CCU (target of approximately 0.25 acre). Cap placement within this CCU will be carefully tracked and monitored. The Remedial Construction Contractor will provide multiple means of measuring the placed thickness (e.g., cores, catch pans, or grade stakes) in addition to bathymetric survey data.

In addition, cores will be collected following placement of the chemical isolation layer for visual inspection and testing to verify that the design GAC concentration has been achieved (see Attachment C for testing SOPs). If necessary, the Remedial Construction Contractor will be required to make refinements to the material blending and/or placement process, and an additional cap placement test will be required to demonstrate achievement of the required results. The Remedial Construction Contractor will also be required to repeat the cap placement test if placement means and methods (the pre-placement blending ratio; the pre-placement GAC content; or the placement means, methods, or rates) are changed during the course of the work or at any time requested by the Construction Manager.

4.5.5 Chemical Isolation Layer Composition Testing

Following successful completion of the Cap Placement Test described in Section 4.5.4, additional GAC testing of the mixed sand for the chemical isolation layer will be performed

on pre-placement samples at a frequency of one sample per 5,000 tons of mixed material, as described in Specification Section 31 23 23 – Capping and Backfilling.

4.6 Floodplain Areas

As part of the Grasse River remediation, excavation of some floodplain areas will be performed. Following verification that excavation has achieved the cleanup criteria, backfill will be placed to return the areas to pre-excavation grade, where feasible, or to a stable grade. The following sections summarize the floodplain area excavation and backfill verification procedures. The following QA/QC steps will be performed:

- **Verification of required grade**: Verification that floodplain excavations have been satisfactorily completed over the required areas and to the required grades
- **Verification of import material quality**: The chemical and physical characteristics of the materials to be used for backfilling will be verified for their intended use (see Section 4.5.1 for details)

4.6.1 Removal Depth Verification

NYSDEC's Division of Environmental Remediation (DER) Technical Guidance for Site Investigation and Remediation (DER-10; NYSDEC, May 2010) provides guidance for soil verification sampling.

The soil verification sampling approach for the floodplain areas has been developed consistent with guidance provided in DER-10. The verification sampling approach for the floodplain areas is as follows:

- Excavation Sidewalls: The excavation limits shown on the Drawings in Appendix A have been delineated using soil sampling locations where previously collected samples achieve the cleanup goals (1 mg/kg). Where collected, samples are typically located within 30 linear feet of each other consistent with the sidewall sampling guidelines presented in DER-10. Therefore, the previously collected samples will serve as the excavation sidewall samples at the "exterior" excavation boundaries. Verification sidewall samples will only be collected to supplement the previously sampled locations in instances where previous sampling density is less than one sample per 30

linear feet. Soil excavation is assumed to be completed to the Grasse River edge of water (elevation 155.5 feet); therefore, verification samples will not be collected from excavation sidewalls along the Grasse River.

- Excavation Bottoms: Consistent with the DER-10 guidance, verification samples will be collected at a frequency of one sample per approximately 900 square feet (sf) from the excavation bottom surface area. At a minimum, one excavation bottom sample will be collected from each excavation area. Verification samples will be collected from the 6-inch depth interval immediately below the excavation bottom and submitted for PCB analysis. Additional sections below 6 inches may be collected and archived for potential analysis pending the results of the surficial samples. If till or bedrock is encountered, excavation bottom samples will not be collected, and no additional excavation will be completed. If oil staining is observed at the base of the excavation, Arconic will work with the Agencies to identify an acceptable path forward based on the site-specific conditions encountered.
- Verification soil samples will be analyzed for PCBs and compared the 1 mg/kg cleanup criteria. If verification sample analytical results indicate that soil cleanup criteria have not been achieved, additional soil excavation will be completed as follows:
  - For excavation sidewalls, the excavation will be extended by up to 10 feet (or as directed by the Engineer) at the given sidewall. Following soil removal, an additional excavation sidewall sample will be collected. This process will be repeated until the soil cleanup criteria have been achieved. If the extended horizontal removal limits expand the excavation bottom by more than 900 sf, an additional excavation bottom sample will be collected from the extended area (i.e., one sample for every additional 900 sf).
  - For excavation bottoms, the approximately 900 sf (or less, depending on removal area size and number of samples) represented by the sample with the exceedance will be excavated up to an additional 6 inches, unless otherwise directed by the Engineer. Following the additional soil removal, an additional verification sample will be collected from the bottom of the excavation.
- This process will be repeated until the 1 mg/kg cleanup criteria has been achieved, unless additional excavation is not practicable (e.g., based on soil stability considerations or the presence of till or bedrock). If additional excavation is not

feasible (e.g., due to encountering till or bedrock or based on geotechnical concerns), the collected verification samples will serve as documentation of the post-excavation conditions (i.e., documentation samples).

4.7 Survey and Positional Control

All equipment used for near shore dredging, main channel capping, post-dredge backfilling, and other remediation-related surveys in the Grasse River will employ hydrographic surveying, engineering, and equipment positioning software. Survey methods for multibeam and single-beam acoustical systems will generally conform to guidelines set forth in the guidance document EM 1110-2-1003, *Engineering and Design-Hydrographic Surveying* (USACE, November 2013). All positioning data for the survey will be based on real-time kinematic (RTK) GPS, which typically provides accuracies of plus 1 centimeter horizontally and plus 4 centimeters vertically.

RTK corrections will be provided from either a project base station via radio link or New York State RTK network depending on network availability. Survey personnel will coordinate with other RTK GPS users on the project and make use of shared resources. All survey data and controls will be referenced to the following:

- Horizontal Datum
  - New York State Plane Coordinate System
  - Eastern Zone (FIPS 3101)
  - North American Datum 1983, 1997, 2011 adjustment
  - Units: United States Survey Feet
- Vertical Datum
  - United States Lake Survey of 1935
  - Units: United States Survey Feet

4.8 Control of Measuring and Test Equipment

Calibration of the equipment will follow the manufacturer's procedure as specified in the Operations and Maintenance Manuals for the test equipment.

4.9 Manual Surveying Method for Delineation of High Subgrade Areas

When a shoreline or other area is encountered with high subgrade or un-dredgeable bank (clay, hard sand/gravel, or rock) that prevents the Remedial Construction Contractor from achieving the design elevations of a DMU, manual surveying will be performed in by the Contractor to determine the extent of this high subgrade area. After the high subgrade area is delineated, a summary figure will be created for review and approval by the Agencies.

4.10 Effluent Discharge Monitoring

Process water generated from the dewatering of sediment during the dredging operations will be monitored at various points during the process and upon ultimate discharge. Sediments will be allowed to gravity drain on scows prior to offload. Cement or other stabilization agents may be added on the scows prior to sediment offload to support bulking and stabilization, as well as to reduce liquids management. Any free water in the scows will be pumped directly to water treatment prior to primary in-barge sediment stabilization. Sediment will then be offloaded to the sediment processing area at the staging area for further dewatering and stabilization, if necessary. Process water generated on the scows and in the sediment processing area will be collected and pretreated at the staging area. The pretreatment process water will be monitored to meet Arconic treatment plant acceptance criteria of 20 parts per million for total suspended solids and 3 parts per billion for PCBs. Pretreated process water from the staging area will then be conveyed through a single-walled, 4-inch, high-density polyethylene force main to Impoundment 005, followed by further treatment for PCBs at Arconic's water treatment plant prior to discharge to the Grasse River at Outfall 004 under Arconic's State Pollutant Discharge Elimination System (SPDES) Permit No. 1732. Discharge monitoring and sampling will be conducted at Outfall 004 in accordance with the frequency and parameters set forth in SPDES Permit No. 1732.

4.11 Water Quality Monitoring

The following sections describe the monitoring program developed through consultation with USEPA, NYSDEC, and SRMT and experience from Grasse River pilot studies, the St. Lawrence River Remediation Program, and other New York State sites. The water quality monitoring plan is included in the Environmental Monitoring Plan (Attachment D), and a summary of the program is provided in Sections 4.11.1 through 4.11.3. Analytical sample

results will be submitted to the selected laboratory for analysis, with results requested on an accelerated turnaround time (TAT) of 24 to 48 hours. Note that sample shipping transport times, the time of day when the sample is collected and submitted, and weekend work may impact the selected laboratory's ability to meet this TAT, and sample results may not be available for 48 to 96 hours after collection.

4.11.1 Monitoring Program Overview and Point of Compliance

During in-river and adjacent floodplain debris removal, dredging, excavation, backfilling, and capping, water column and water intake monitoring will be performed. Locations for water column monitoring include a fixed location upstream (upstream of RA areas), a near-field station (mobile station approximately 1,000 feet downstream of the work areas), a fixed location established at the historic water column sampling location WC013 (located just upstream of the Grasse River mouth at T71), and a far-field station in the St. Lawrence River. At each station, monitoring will be performed at the midpoint of the river near the water surface and the mudline (i.e., 0.2 and 0.8 times the total water column depth) to account for stratification. Grab samples will be composited if stratification is not present. If water depths are shallow (e.g., approximately 5 feet or less), monitoring will be performed mid-depth (0.5 times the total water column depth).

The Arconic East Plant and SRMT water intakes will also be sampled at the ports within each facility.

4.11.2 Water Quality Monitoring During Dredging and Backfilling

During debris removal, dredging, excavation and post-removal backfilling, the monitoring program includes monitoring for total suspended solids (TSS) and PCBs (Aroclor) in the water column, as well as turbidity and PCBs (Aroclor) at the Alcoa East Plant and SRMT water intakes. Turbidity monitoring will be a daily single real-time reading for the water intakes. Water column samples will be collected as daily composites at the various depths as applicable, and water intakes will be daily grab samples from the sampling ports. Monthly sampling for PCB congener analysis will also be performed for the SRMT water intake. See the Environmental Monitoring Plan (Attachment D) for additional details on frequency. Water quality monitoring will rely on numeric compliance standards rather than visual

criteria. TSS and PCB results will be requested on an accelerated TAT (request 24 hours, but results may not be available for 48 to 72 hours).

Grab samples will be collected once a week for the first month of near shore dredging, debris removal, and backfilling from the near-field station and submitted for analysis of lead, mercury, and select polycyclic aromatic hydrocarbons (PAHs). Results will be requested on an accelerated TAT.

Advisory and corrective action levels will apply as described below. The levels include the following:

- **TSS (Advisory Level)** – 100 milligrams per liter (mg/L) over ambient (upstream location) at the near-field in-river station
- **Turbidity (Corrective Action Level)** – 100 Nephelometric Turbidity Units (NTUs) above baseline conditions at the water intakes
- **PCB (Advisory Level)** – 0.5 microgram per liter (µg/L) at the near-field and T71 in-river stations
- **PCB (Corrective Action Level)** – 0.5 µg/L at the St. Lawrence River in-river station and at the intakes
- **Metals (Advisory Level)** – Less than standard specified in 6NYCRR Part 703.5 for Class C (chronic aquatic);[2] assessed by comparing the sample result at the near-field station against the levels for the following:
  - **Lead (dissolved)** – 2.3 µg/L
    - Lead Formula = {1.46203 – [ln (hardness) (0.145712)]} exp (1.273 [ln (hardness)] – 4.297)
    - Hardness determined as an average of the means from Reaches 4 through 8 in the River and Sediment Investigation (RSI) Phase I – 63 mg/L
  - **Mercury (dissolved)** – 0.77 µg/L

[2]Available at: https://govt.westlaw.com/nycrr/Document/I4ed90418cd1711dda432a117e6e0f345?viewType=FullText&originationContext=documenttoc&transitionType=CategoryPageItem&contextData=(sc.Default))

- **PAHs** – Less than the standard specified in the New York State Division of Water Technical and Operational Guidance Series 1.1.1 for Class C (acute aquatic);[3] assessed by comparing the sample result at the near-field station against the level
  - **Anthracene** – 35 µg/L
  - **Benz(a)anthracene** – 0.23 µg/L
  - **2-Methylnaphthalene** – 42 µg/L
  - **Naphthalene** – 110 µg/L
  - **Phenanthrene** – 45 µg/L
  - **Pyrene** – 42 µg/L

Exceedances of the numeric levels will result in the actions listed below. These actions are described further in the Contingency Plan (Appendix L of the FDR).

- Review results to confirm the exceedance is project-related or resample for confirmation of results
- Perform additional monitoring as necessary to investigate the cause of the exceedance
- Review work operations and best management practices
- Identify possible operational adjustments or BMPs (to the extent practicable) to reduce the concentrations in the water column
- Identify possible in-river operations adjustments if results are confirmed

If approved by USEPA, NYSDEC, and SRMT, adjustments to the sampling frequency and parameter list may be made based on the results of monitoring during the initial phases of work for each operation.

4.11.3 Water Quality Monitoring During Capping

Water quality monitoring activities during capping include TSS monitoring at the upstream, near-field (mobile), T71 (mouth), and far-field (St. Lawrence River) stations and at the water intakes as described in Section 4.11.2. Grab samples will also be collected once a day during the first month of capping activities from monitoring stations downstream of capping activities and submitted for PCB (Aroclor) analysis. The same action levels listed in

[3] Available at: http://www.dec.ny.gov/docs/water_pdf/togs1112.pdf

Section 4.11.2 will apply. If approved by USEPA, NYSDEC, and SRMT, adjustments to the sampling frequency and parameter list may be made based on the results of monitoring during the initial phases of work for each operation.

4.12 Air Quality Monitoring

Air quality monitoring will be conducted during near shore debris and sediment removal, floodplain excavation, contaminated sediment dewatering and processing, capping and backfill operations, and landfill operations.

The air monitoring program includes particulate and VOC measurements and PCB analysis. PCB analysis will be conducted during near shore debris and sediment removal, floodplain excavation, contaminated sediment dewatering and processing, and landfill operations; it will not be conducted during backfill or capping only operations. Particulate and VOC monitoring will be performed in the vicinity of sources of potential particulate emissions during intrusive activities including grading, sediment and soil handling and processing at the staging area, and disposal at the SLF. Particulate monitoring will be performed during backfill and cap material handling at the staging area. Monitoring includes using real-time meters for particulate matter less than 10 microns (PM_{10}) and VOCs to obtain continuous readings for determining a continuous 15-minute average. Continuous 24-hour air samples will be collected daily and submitted for PCB (Aroclor) analysis on an accelerated TAT (request 48 hours, but results may not be available for 72 to 96 hours after collection).

Air monitoring locations will be established surrounding the dredging/excavation areas (dredge corridor), sediment processing area (staging area), and SLF. The upwind and downwind locations will be determined each day in consideration of the typical prevailing wind direction and through observation of meteorological conditions and data (e.g., wind speed and direction, wind gust, temperature, and humidity). Wind data will be obtained from the local Massena, Federal Aviation Administration Airport Meteorological Station.

PM_{10} and VOC corrective action levels will apply at the downwind location only, and PCB levels apply to all stations. The proposed corrective action levels include the following:

- PM_{10} – 0.150 milligram per cubic meter above the upwind location during a 15-minute period
- VOC – 25 parts per million above the upwind location during a 15-minute period
- PCB (Aroclor) – 0.100 microgram per cubic meter

These corrective action levels have been developed in consideration of the New York State Department of Health Generic Community Air Monitoring Plan provided in DER-10 (NYSDEC, May 2010) and the levels specified during the Remedial Options Pilot Study by USEPA. Exceedance of the corrective action levels will result in corrective action as described in the Contingency Plan (Appendix L of the FDR) and may include a review of work activities for any obvious causes, continued readings, and modifications to remediation activities.

If approved by USEPA, NYSDEC, and SRMT, adjustments to the sampling frequency and parameter list may be made based on the results of monitoring during the initial phases of work for each operation.

4.13 Noise Monitoring

Noise during operations will vary throughout the day and evening hours and will primarily be managed using mufflers and silencers on equipment where possible. The staging area will contain the largest concentration of equipment with potential for noise impacts, including various generators, pumps, transport trucks, and backup alarms. The specifications developed for the work (Appendix B of the FDR) provide the requirements for the management and reduction of noise generation, including the use of critically silenced or hospital-grade generators and pumps, use of hardwired power where possible to reduce the need for generators and engines, and the reduction of some activities during evening hours to lessen generated noise.

The staging area will be configured considering the potential for noise generation. The staging area will use electrical motors and equipment where practical. Efforts will be made to locate equipment and noise sources away from potential receptors or shelter operations to reduce impacts. The existing topography, including the Route 131 bridge approach grading,

will assist in sheltering some operations from the adjacent public. Efforts to reduce noise during on-water operations will be focused on installation of control measures on equipment, including the use of silencers and mufflers on barge-based equipment.

It is anticipated that a noise monitoring program will be performed for 1 day at the start of a new construction activity (e.g., start of dredging or armored capping) to assess levels. The program will include the use of a real-time Type 1 or Type 2 sound level meter as rated by the American National Standards Institute to measure levels between the construction activities and the nearest receptor. If levels are acceptable at the community receptor location, routine noise monitoring will be performed monthly to assess overall project levels, and no other specific additional monitoring would be performed until a new activity begins or the mode of operation changes significantly. Changes in the mode of operations during construction will be determined in coordination between the remedial contractor and Construction Manager. This decision will be made by considering the equipment to be used, any modifications to be made, the activities to be performed, and the proximity to receptors. Any change in construction methods that will result in an increase in noise will require noise monitoring. The remedial contractor and Construction Manager will discuss the overall process and potential impacts to noise levels to assess the need for additional noise monitoring; this decision will be reviewed with USEPA, NYSDEC, and SRMT. Records of the measurement—including specifics of the measurement location, time of measurement, meteorological conditions at the time, identification of significant sound sources, model, serial number, and calibration of equipment—will be maintained during all monitoring events.

The Town of Massena does not have regulations or requirements regarding allowable noise levels. As such, sound levels at the perimeter of the site will be evaluated against the criteria provided in this section. These criteria have been developed considering a reasonable range of sound levels with the intent of minimizing nuisance noise. The following criteria are expressed in A-weighted decibels (dBA):

- Residential receptor – 80 dBA in the daytime (maximum hourly average; 7 a.m. to 10 p.m.) and 65 dBA in the evening (maximum hourly average; 10 p.m. to 7 a.m.)
- Commercial receptor – 80 dBA (maximum hourly average)

Meters will run for a full hour every 4 hours over the 24-hour period to assess the corrective action levels (e.g., meters will run from 8 to 9 a.m., 12 to 1 p.m., 4 to 5 p.m., 8 to 9 p.m., 12 to 1 a.m., and 4 to 5 a.m.). If construction operations are conducted less than 24 hours per day, the monitoring duration that day will be reduced to only occur during active construction activities. Exceedance of the corrective action levels will result in corrective action as described in the Contingency Plan (Appendix L of the FDR) and may include a review of work activities for any obvious causes and employment of noise mitigation techniques.

5 REPORTING PROCEDURES

5.1 Documentation

A document control system will be established, as described in Section 3.3.5, to provide measures for the control of issuing, distribution, storage, and maintenance of quality-related documents, including documents from subcontractors, off-site fabricators, laboratory suppliers, vendors, and other suppliers.

Preparation, review, approval, and issuance of documents (including revisions) affecting quality will be controlled to the extent necessary to determine that the documents include the specified requirements and provide adequate procedures or guidelines to perform the intended activities. Such documents may include the following:

- Drawings and specifications
- Calculations
- Procedures
- Plans
- Reports
- Design Change Notices
- Field Change Requests

The Project Field Engineer/Construction QA Officer or a designee will review the documents to verify the inclusion of appropriate quality requirements. The Project Field Engineer/Construction QA Officer will be responsible for the maintenance of records providing factual evidence that required QC activities or tests have been performed.

5.2 Construction Quality Control Report

A Construction Quality Control (CQC) Report will be prepared on a regular basis and will document the results of construction activities, inspections, testing, and QC activities performed during RA on the project. The CQC Report will include the following information:

- Significant and unusual occurrences
- Phases of construction in progress
- Material and equipment delivered to or leaving the site

- Weather conditions and their impact on construction activities
- Test and control activities performed, with results and deficiencies noted along with corrective actions
- References to test reports and data
- References to relevant documents (specifications and drawings)
- Submittals and deliverables reviewed, who reviewed them, and relevant action taken
- Off-site surveillance activities, including actions taken
- Description of non-conformance or identified deficiencies
- Instructions given or received
- Changed conditions, delays, or conflicts encountered and directives given that changed the existing performance requirements

Reports will be signed and dated by the Project Field Engineer/Construction QA Officer and provided to USEPA. The CQC Report will include copies of test reports, as well as copies of reports prepared by QA/QC personnel.

5.3 Daily Activities Report

A Daily Activities Report will be prepared for each day that work is performed on site. The records will include the work of the Remedial Construction Contractor, subcontractors, and suppliers and provide the following information:

- Personnel and hours for each day spent on site
- Operating plant and equipment with hours worked, idle, or down for repair
- Work performed each day, including location, description, and personnel involved
- Quantity of materials received at the site with a statement as to acceptability and storage, as well as references to specifications and engineered plan drawings requirements
- Job safety evaluations stating what was checked, results, and instructions or corrective actions

5.4 Storage of Records

Records will be prepared to furnish documentary evidence of the quality of items, services, environmental processes, and engineered systems provided. The records will be readily

retrievable for review and auditing purposes. The records will be controlled in a manner that prevents loss, damage, or other detrimental conditions of the records. Duplicate records will be maintained in separate storage facilities for a period of 10 years after USEPA's notice of completion.

6 PROCEDURES FOR TRACKING DEFICIENCIES AND CORRECTIVE ACTIONS

6.1 Non-conformance Report

Work or materials not conforming to the specifications will be identified by the Project Field Engineer/Construction QA Officer and documented on a Non-conformance Report. The Non-Conformance Report will include the following:

- Description of non-conforming item or activity
- Detailed description of non-conformance
- Referenced criteria
- Recommended disposition and corrective action to prevent recurrence (as applicable)
- Affected organization

The Non-conformance Report will remain open until the non-conforming condition has been satisfactorily resolved and verified by QA personnel.

6.2 Control and Segregation

The non-conforming materials or items will be controlled to prevent inadvertent use or further processing. Items determined to be non-conforming will be identified and segregated from acceptable items.

6.3 Disposition

The Non-conformance Reports will include the actions required to bring the non-conforming condition to an acceptable condition and may include reworking, replacing, retesting, or re-inspecting. Implementation of the disposition may be done in accordance with the original procedural requirements, a specific procedure, or instruction.

6.4 Corrective Actions

In addition to resolving identified non-conforming conditions, the corrective action record will also address the initial cause of adverse conditions and establish methods and controls to prevent recurrence of the same or similar types of non-conformance. The Project Field Engineer/Construction QA Officer will track the non-conformance and corrective actions to identify any trends in the causes of the non-conforming conditions and then initiate the

actions necessary to prevent recurrence. The Project Field Engineer/Construction QA Officer will also monitor the corrective actions to verify that they were properly implemented and accepted and that the Non-Conformance Report was completed.

7 DATA VERIFICATION AND VALIDATION

7.1 Data Verification

Data verification will be performed for all sampling activities, as summarized in Table 1.

Table 1
Data Verification Procedures

Records Reviewed	Required Documents	Description	Responsible Person
Field records	CQAP/ Field SOP	Verify conformance to approved sampling and field measurement procedures, confirm activities met performance criteria, and verify deviations from procedures or criteria were documented.	Project Field Engineer/ Construction QA Officer or designee
Analytical data deliverables, contractual documents	CQAP	Verify that the required deliverables, analyte lists, method holding times, analytical procedures, laboratory qualifiers, measurement criteria, and project QLs conform to specifications. Verify that deviations from procedures or criteria were documented.	Project Field Engineer/ Construction QA Officer or designee
Field records, database output	CQAP/ Field SOP	Verify transcription of field data from field forms to database.	Project Field Engineer/ Construction QA Officer or designee
Custody records, analytical data reports	CQAP	Review traceability from sample collection through reporting.	Project Field Engineer/ Construction QA Officer or designee
Laboratory EDDs, analytical data reports, database output	CQAP	Verify a subset of EDD entries against hardcopy or PDF analytical reports.	Project Field Engineer/ Construction QA Officer or designee
Data validation reports, database output	CQAP	Verify that entry of qualifiers was correct and complete.	Project Field Engineer/ Construction QA Officer or designee

Records Reviewed	Required Documents	Description	Responsible Person
Analytical data reports	CQAP	Verify that reported analytes, holding times, analytical procedures, measurement criteria, and project QLs conform to the CQAP. Verify that deviations from procedures or criteria were documented.	Project Field Engineer/ Construction QA Officer or designee
Analytical data reports	CQAP/NFG	100% of the laboratory data will be validated (see details below).	Project Field Engineer/ Construction QA Officer or designee
Analytical data reports	CQAP/NFG	Verify that the qualifiers applied during validation were in conformance with the CQAP and specified validation guidance.	Project Field Engineer/ Construction QA Officer or designee
Data validation reports	CQAP/NFG	Verify data validation was performed in accordance with the CQAP specifications and all required peer reviews were conducted. If validation actions deviated from the CQAP specifications or regional validation guidance based on professional judgment, verify rationale was documented.	Project Field Engineer/ Construction QA Officer or designee
Acoustical bathymetric survey data	Contractor Work Plan	Verify that equipment calibrations and checks have been performed for vertical and horizontal survey measurements	Project Field Engineer/ Construction QA Officer or designee
Manual survey	Contractor Work Plan	Verify that equipment calibrations and checks have been performed for vertical and horizontal survey measurements	Project Field Engineer/ Construction QA Officer or designee
Sample position data	SOP	Verify that equipment calibrations and checks have been performed for horizontal position measurements	Project Field Engineer/ Construction QA Officer or designee
Material quantity tracking records	CQAP	Verify that material quantity records are accurate through independent observations and/or measurements	Project Field Engineer/ Construction QA Officer or designee

Notes:
EDD = electronic data deliverable
CQAP = Construction Quality Assurance Plan
NFG = National Functional Guidelines
QL = quantitation limit
SOP = Standard Operating Procedure

7.2 Data Validation

For all parameters, full validation (Stage 4; USEPA, January 2009) will be performed on the initial sample delivery group (SDG) for each analytical method, matrix, and laboratory. If errors or problems are encountered during full validation, additional Stage 4 validations may be requested.

All other SDGs will receive Stage 2A validation. Stage 2A validation is based on information summarized by the laboratory on its QC forms but includes no raw data review. At a minimum, Stage 2A validation will include the following data elements:

- Agreement of analyses conducted with chain-of-custody requests
- Holding times and sample preservation
- Results of laboratory, equipment, field, and trip blanks
- Surrogate recoveries
- Laboratory control sample and laboratory control sample duplicate results
- Matrix spike and matrix spike duplicate results
- Laboratory duplicate results
- Field duplicate results
- Total solids

Qualifiers will be applied based on the criteria in this CQAP, USEPA National Functional Guidelines (NFG) for data review (USEPA, October 1999, October 2004, June 2008), or professional judgment. Reports summarizing data qualification as a result of the validation effort will be prepared.

For non-analytical data, equipment calibration will be used for validating the accuracy of the collected data. Equipment calibrations will be performed in accordance with manufacturer recommendations.

As data validation and verification tasks proceed and are completed, project quality objective and data quality objective evaluations will be performed throughout the duration of the project, and the usability of the data in meeting the stated project objectives will be continually assessed by the appropriate technical staff. Any limitations on the use of data

collected for the project will be conveyed in writing to the Construction Project Manager and all applicable users in a timely manner for potential impact analysis. Resolution and reconciliation of all issues raised by the data users will be coordinated and documented by the Construction Project Manager. If warranted, any corrective actions taken will be documented by the Construction Project Manager (or designee) and approved by the Agencies prior to corrective action implementation.

8 SCHEDULE

It is currently estimated that the near shore dredging, main channel capping, and post-dredge backfilling will be conducted throughout four construction seasons, beginning with a Staged Cap Test that was performed in fall 2018 and full-scale work from spring 2019 to fall/winter 2022. The construction season will generally run from April to November of each year, with some fluctuations allowed for weather and river conditions. It is expected that in-water construction operations may be conducted up to 24 hours per day and up to 6 days per week, with the final schedule to be decided by the selected Remedial Construction Contractor. The schedule will be reviewed and updated as necessary prior to construction each year.

9 REFERENCES

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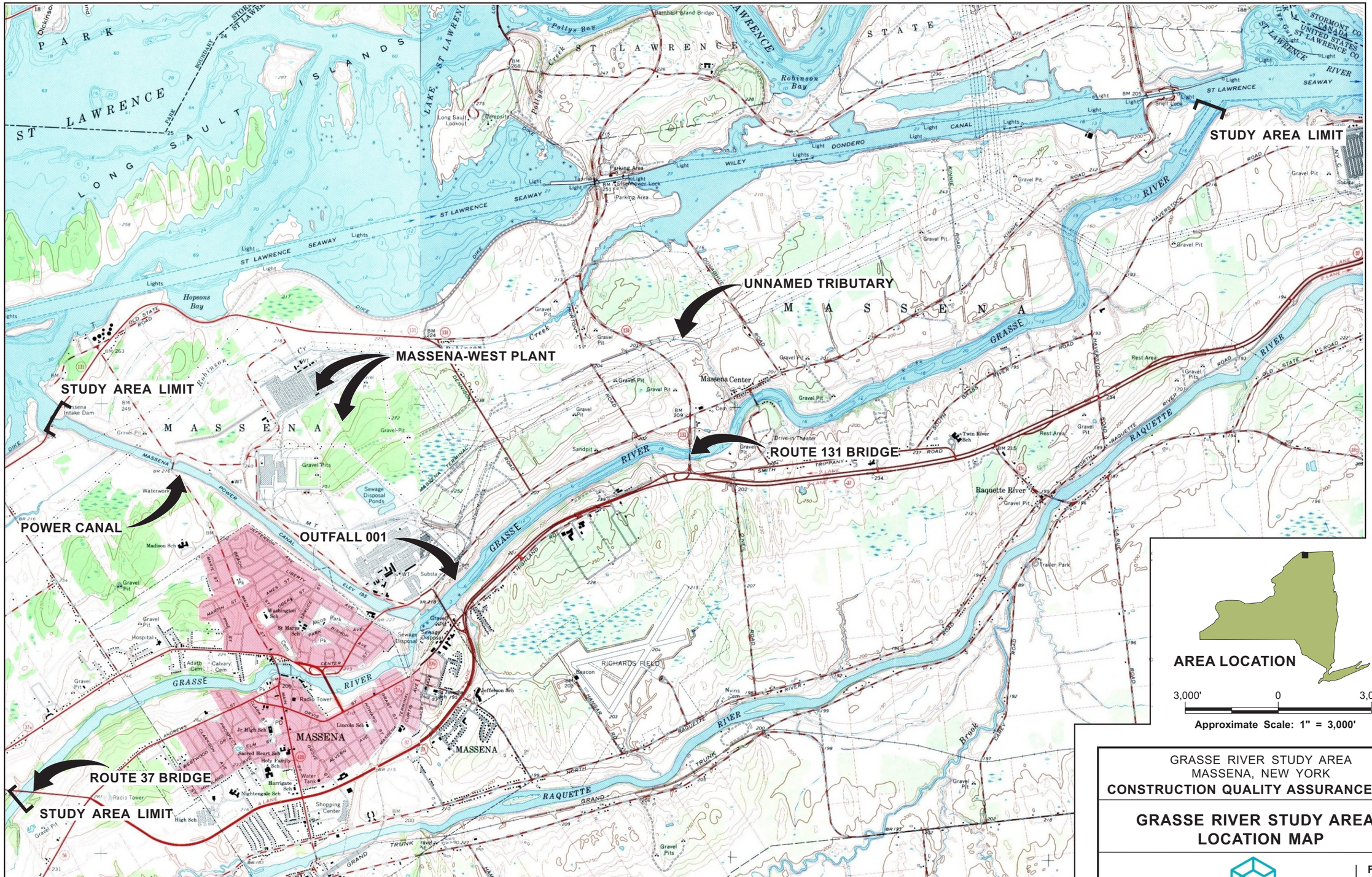
FIGURES



GRASSE RIVER STUDY AREA
MASSENA, NEW YORK
CONSTRUCTION QUALITY ASSURANCE PLAN

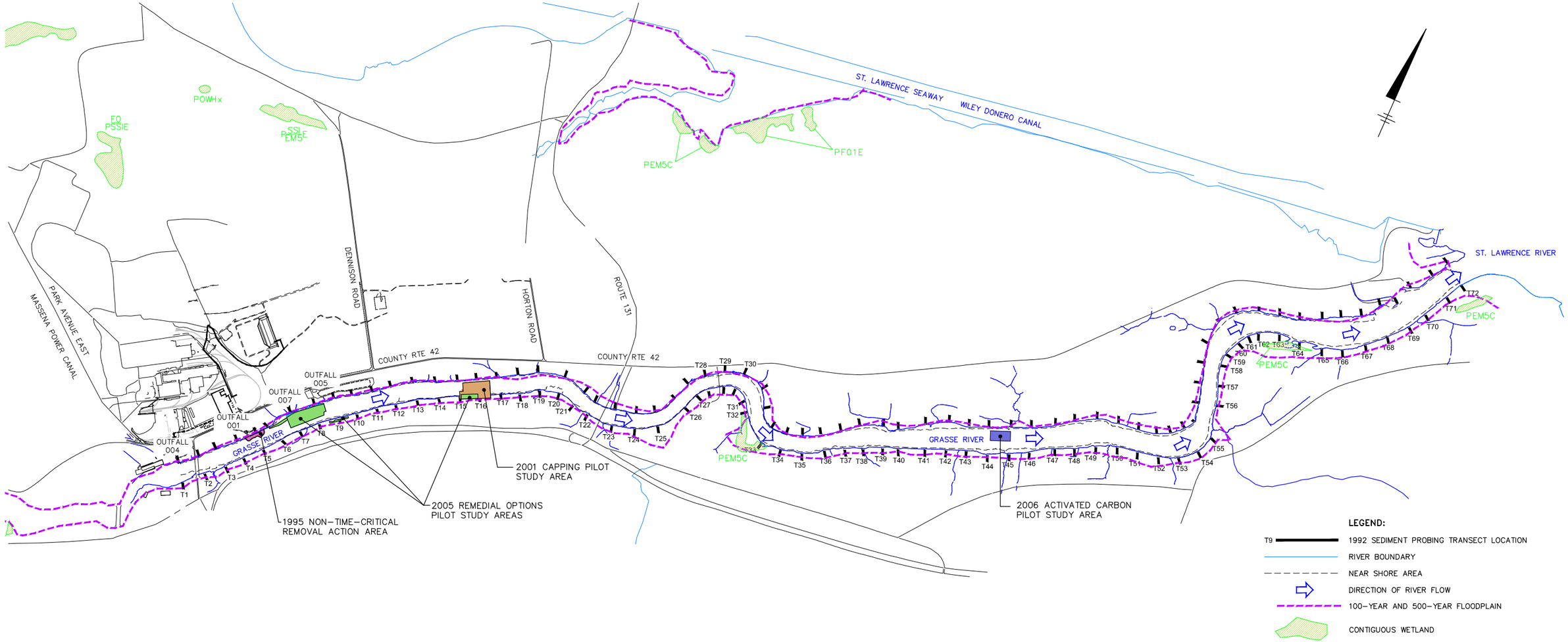
GRASSE RIVER STUDY AREA - LOCATION MAP

ARCONIC

FIGURE
C1-1

10/11/2017 SYRACUSE, NY-ENV/CAD-DJHOWES, K.SARTORI
B0010886/0032/00001/CDR/10886N02.CDR

REFERENCE: MASSENA, NEW YORK-ONTARIO USGS QUAD. 1964 RAQUETTE RIVER, NEW YORK-ONTARIO USGS QUAD. 1964



WETLAND LEGEND

ECOLOGICAL SYSTEM	P-PALUSTRINE			
SUBSYSTEM	NONE	NONE	NONE	NONE
CLASS	EM EMERGENT	SS SCRUB/SHRUB	FO FORESTED	OW OPEN WATER/ UNKNOWN BOTTOM
SUBCLASS	5 NARROW-LEAVED PERSISTENT	1 BROAD-LEAVED DECIDUOUS	1 BROAD-LEAVED DECIDUOUS	

MODIFYING TERMS

WATER REGIME	SPECIAL MODIFIERS
NON-TIDAL	x EXCAVATED
C SEASONAL	
E SEASONAL SATURATED	
H PERMANENT	

NOTES:

1. BASE MAP TAKEN FROM PLANIMETRIC MAPPING PREPARED BY LOCKWOOD MAPPING, INC. USING 11/9/92 AERIAL PHOTOGRAPHY. RIVER OUTLINE EXTENDING FROM THE RT. 37 BRIDGE APPROXIMATELY 6000 FEET DOWNSTREAM (SOUTHWEST GRASSE RIVER STRETCH) DERIVED FROM BASEMAP PREPARED BY ECOLOGY AND ENVIRONMENT, INC. AND SHOULD BE CONSIDERED APPROXIMATE ONLY.
2. EXTENT OF NEAR SHORE AREAS PROVIDED BY QUANTITATIVE ENVIRONMENTAL ANALYSIS, LLC (QEA). ST. LAWRENCE RIVER WATERLINE GENERATED USING 5/1/01 AERIAL PHOTOGRAPHY.
3. EXTENT OF FLOODPLAIN DIGITIZED FROM FLOOD INSURANCE RATE MAP (TOWN/VILLAGE OF MASSENA, NEW YORK, ST. LAWRENCE COUNTY) COMMUNITY - PANEL NUMBERS 360705 0001 B (FEMA, NOVEMBER 1980) AND 361182 0025 B (FEMA, JUNE 1986) AND IS SHOWN IN FOR THE GRASSE RIVER, THE MASSENA POWER CANAL AND ROBINSON CREEK ONLY.
4. WETLANDS DIGITIZED FROM NATIONAL WETLANDS INVENTORY (USDOI) MAPS 502 AND 709. DEPICTED WETLANDS REPRESENT THOSE WETLANDS CONTIGUOUS TO THE MASSENA POWER CANAL, ROBINSON CREEK, AND THE LOWER GRASSE RIVER ONLY.

0 1200' 2400'
GRAPHIC SCALE

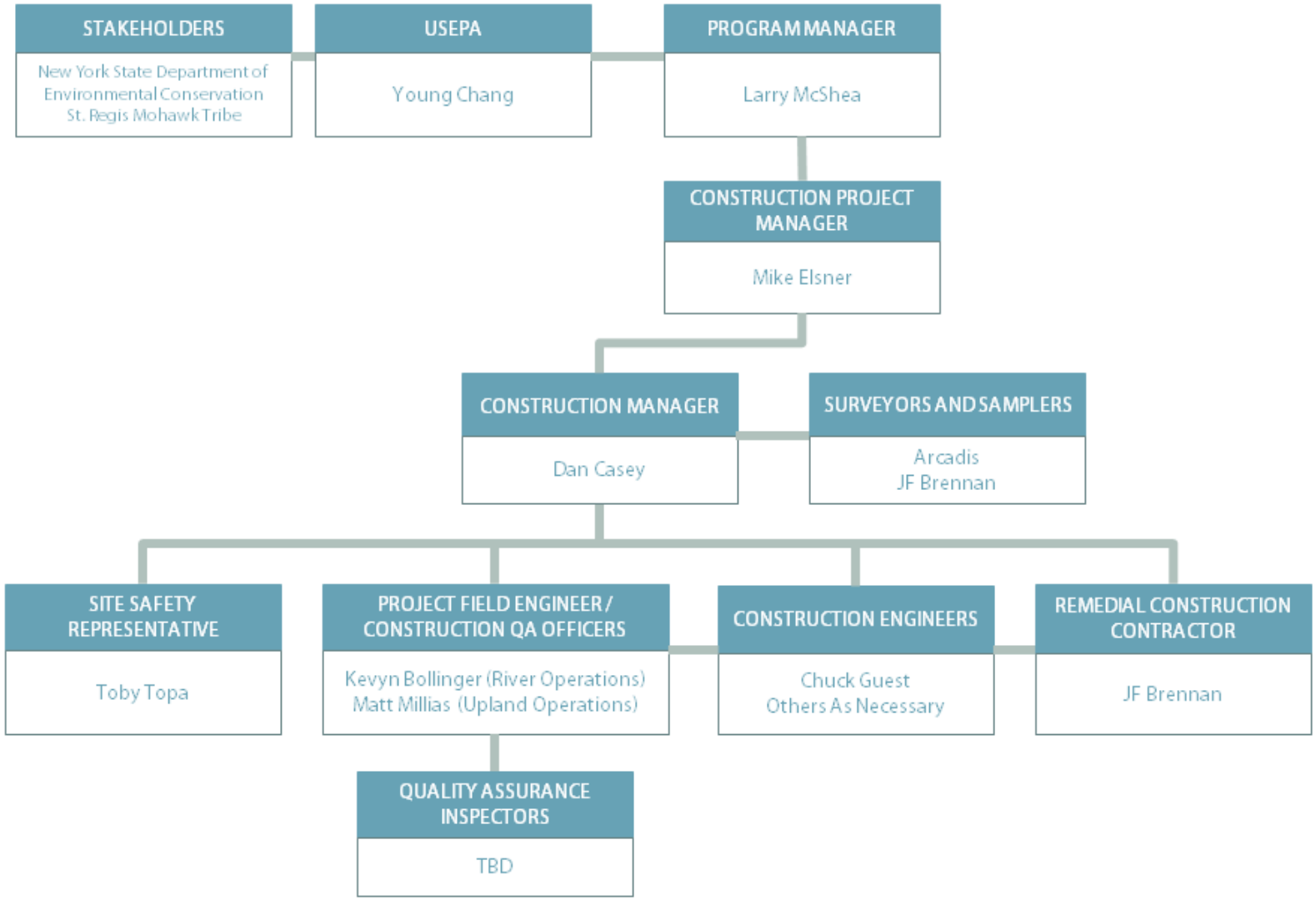
GRASSE RIVER STUDY AREA
MASSENA, NEW YORK
CONSTRUCTION QUALITY ASSURANCE PLAN

SITE MAP

ARCONIC

FIGURE
C1-2

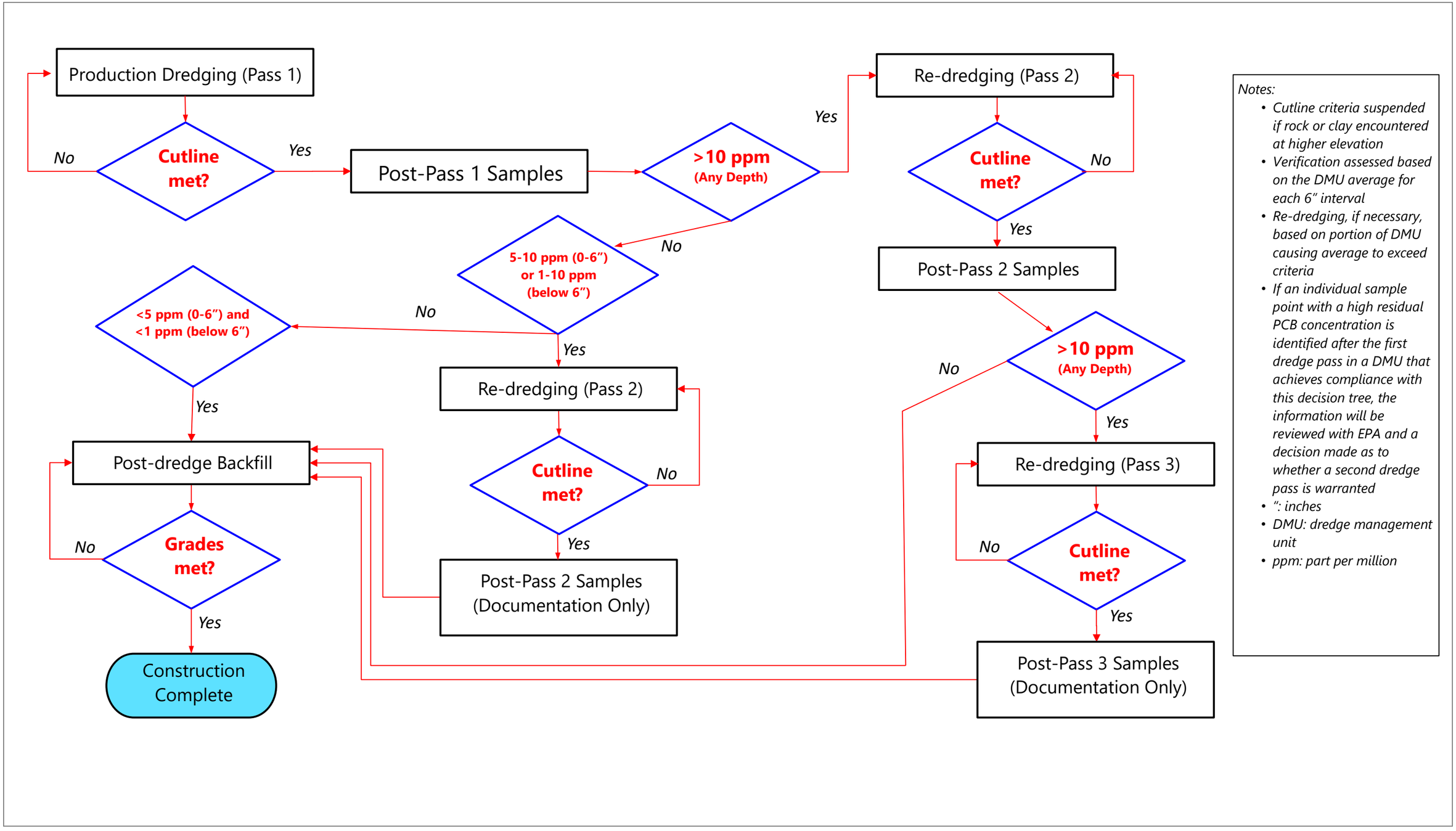
CITY: SYRACUSE, NY DIV/GROUP: IMDV/CAD DB: K.DAVIS, K.SARTORI PIC/PM: H. VANDEWALKER TM: S. HILL LYR: ON=*; OFF=*REF*
G:\ENVCAD\SYRACUSE\ACT\B0010886\00350\0001\CQAP-REPORT\B0010866B02.dwg LAYOUT: C1-2 SAVED: 10/11/2017 12:59 PM ACADVER: 20.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: PLTFULL.CTB PLOTTED: 10/11/2017 12:59 PM BY: SARTORI, KATHERINE
XREFS: 10876X02 10886X00
IMAGES: Arconic.jpg



Filepath: http://portal/sites/GrasseRiverPCB/Document Library/Remedial Design/Final Design/Final Design Report - April 2019/Appendix C - CQAP/Figure_C3-1_Org Chart.docx

ARCONIC

Figure C3-1
Organization Chart
Construction Quality Assurance Plan
Grasse River Project/Arconic



Filepath: http://portal/sites/GrasseRiverPCB/Document Library/Remedial Design/Final Design/Final Design Report - April 2019/Appendix C - CQAP/Figure_C4-1_DredgeVer_DecisionTree_June2019.docx

ARCONIC

Figure C4-1
Dredge Verification and Backfill Decision Tree
Construction Quality Assurance Plan
Grasse River Project/Arconic

Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N01
- DMU-N03
- DMU-S02

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.

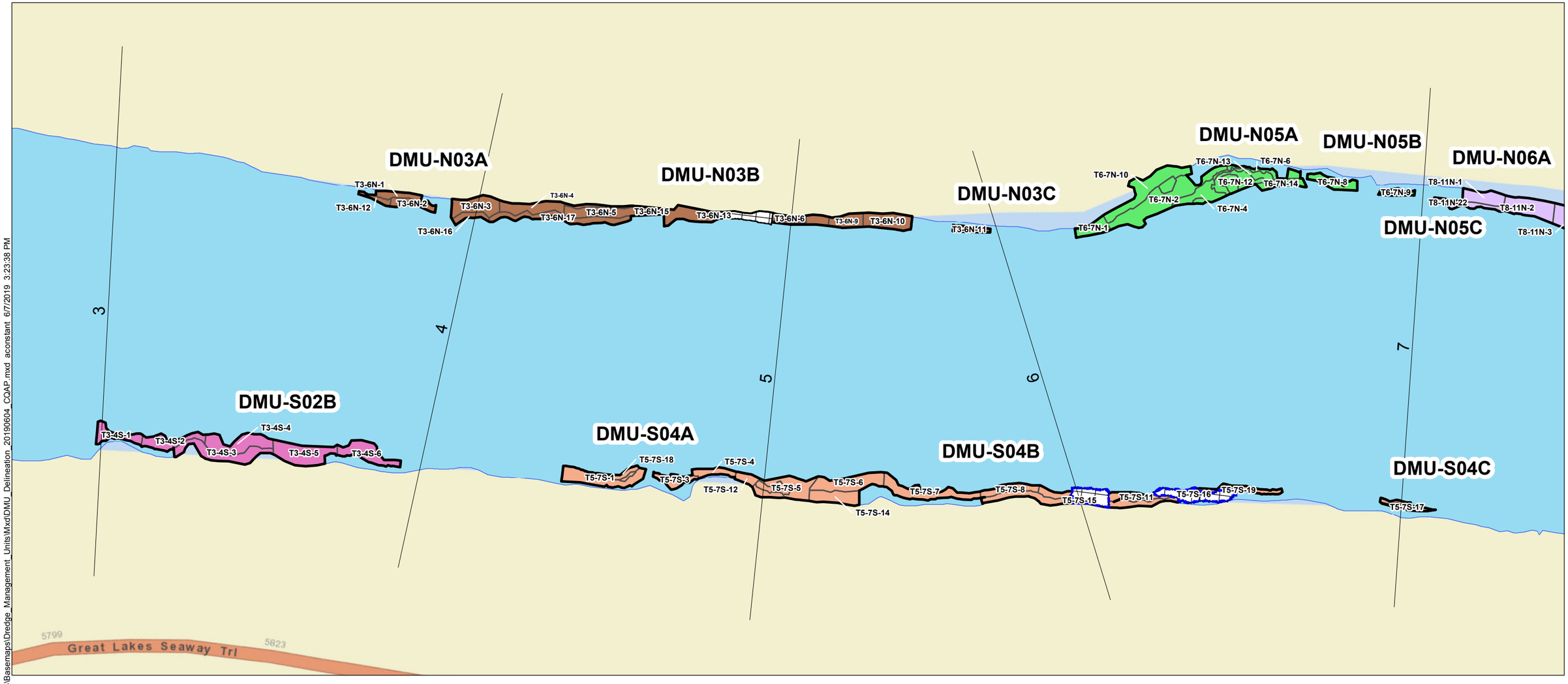
DMU = Dredge Management Unit

Feet
0 100 200

ARCONIC

Figure CA-1
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:23:28 PM



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N03
- DMU-N05
- DMU-N06
- DMU-S02
- DMU-S04

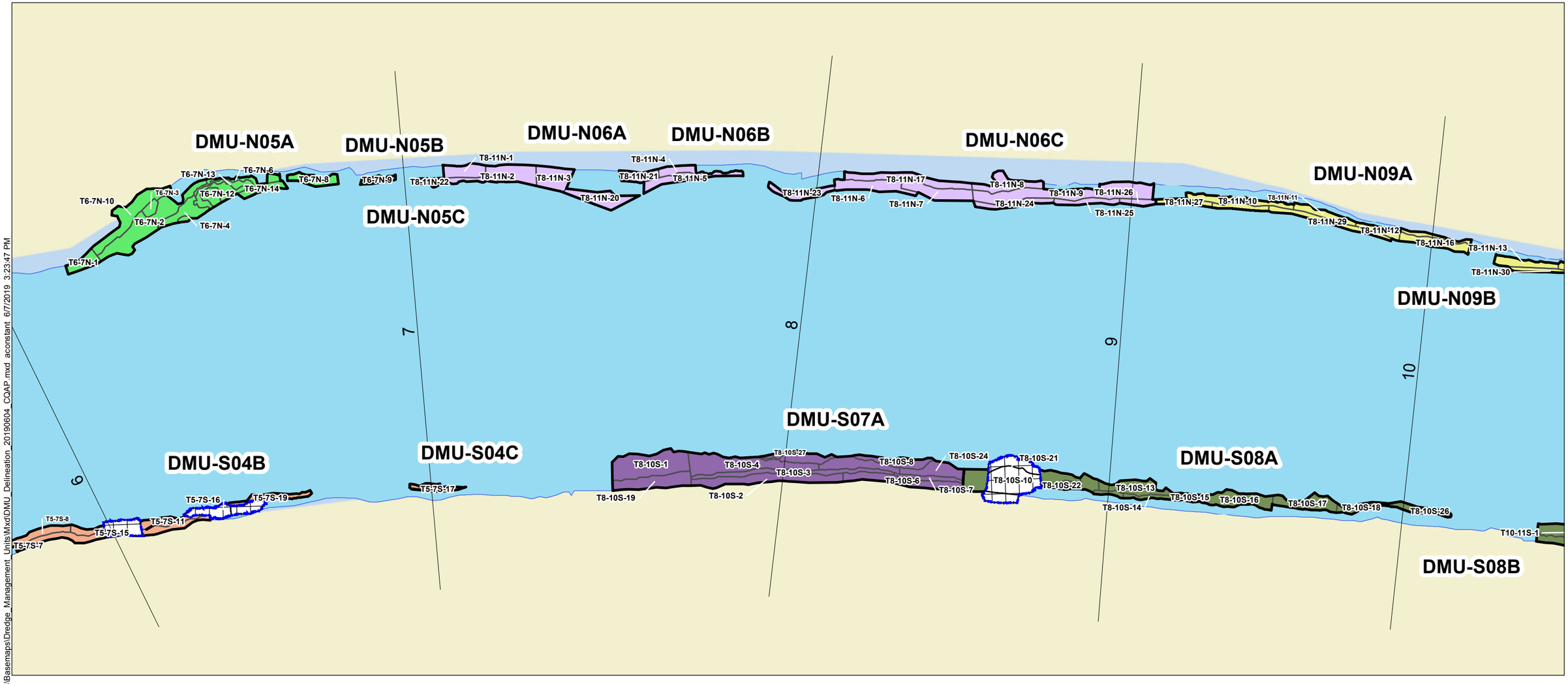
NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:23:38 PM

ARCONIC

Figure CA-2
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N05
- DMU-N06
- DMU-N09
- DMU-S04
- DMU-S07
- DMU-S08

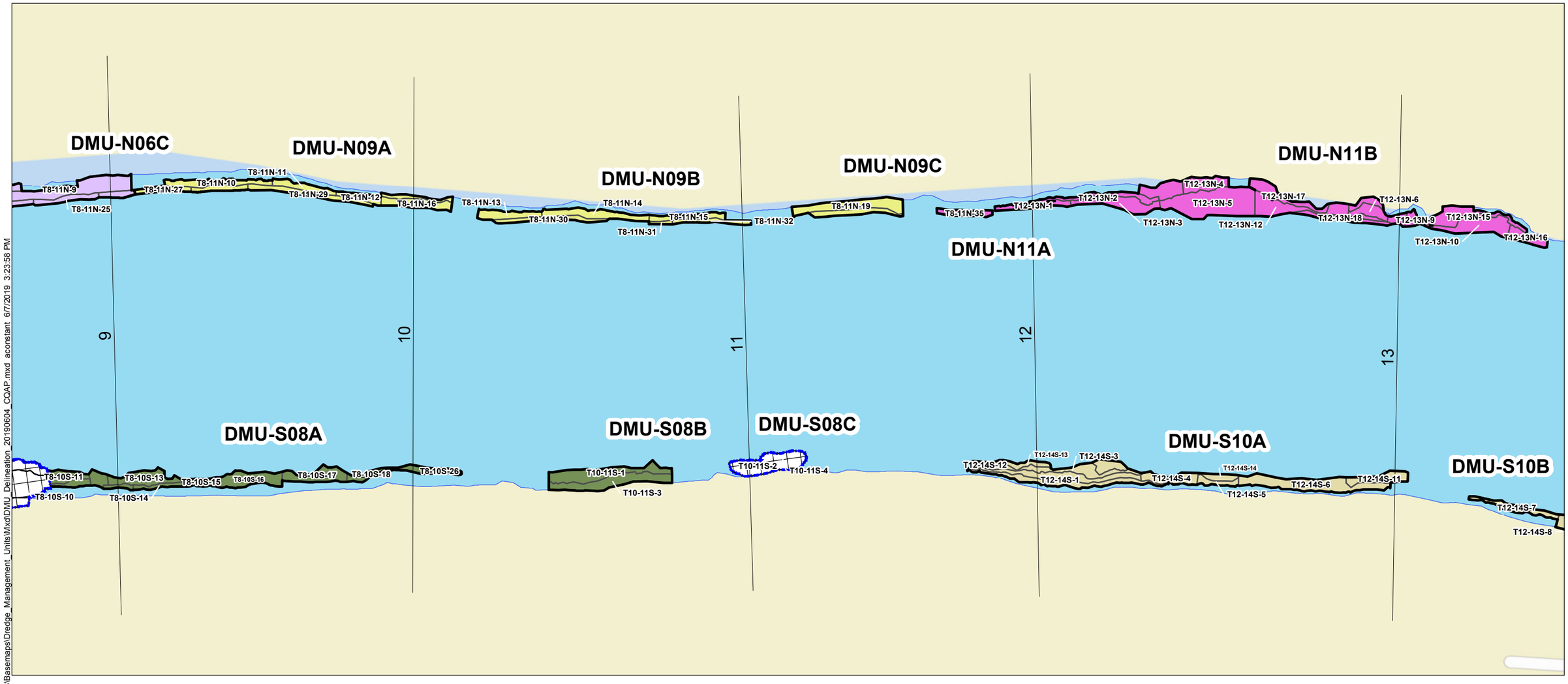
NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

ARCONIC

Figure CA-3
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:23:47 PM



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N06
- DMU-N09
- DMU-N11
- DMU-S08
- DMU-S10

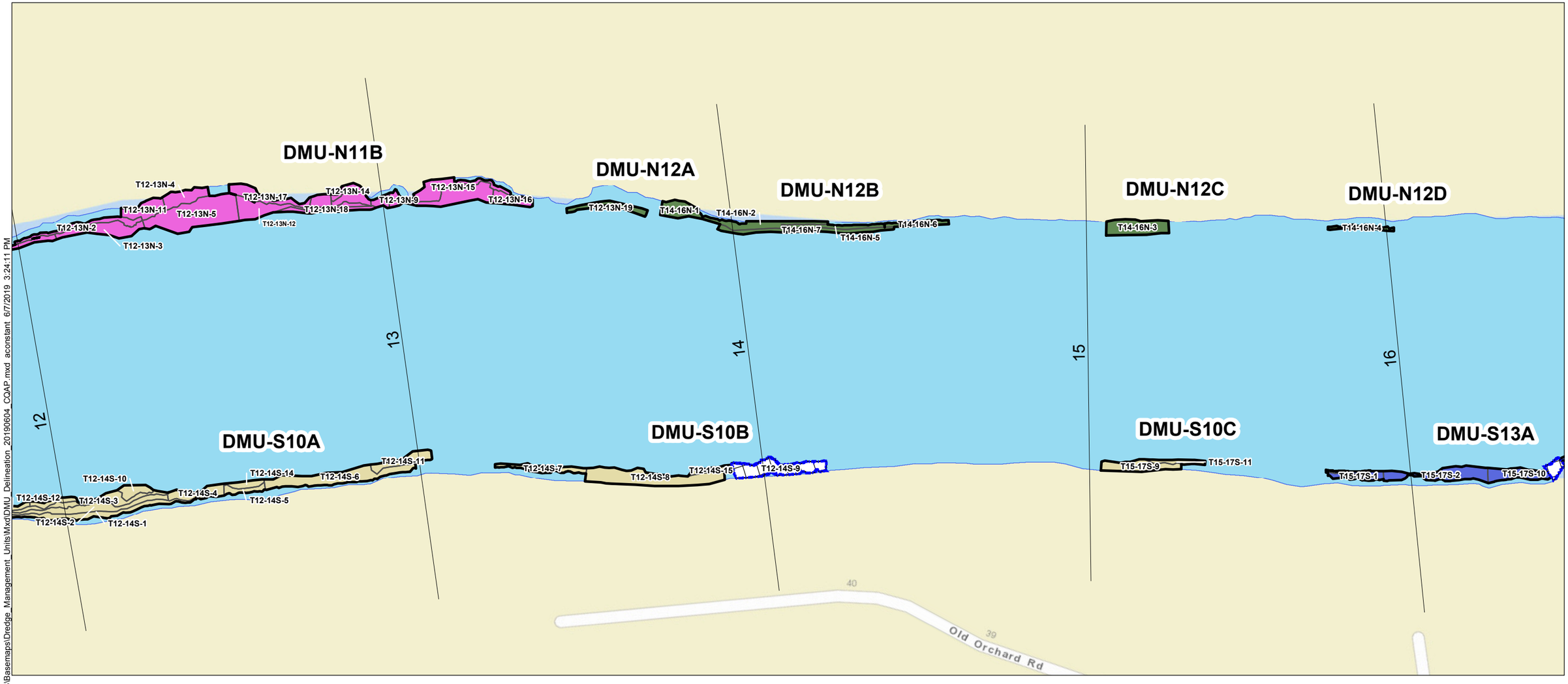
NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

ARCONIC

Figure CA-4
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:23:58 PM



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N11
- DMU-N12
- DMU-S10
- DMU-S13

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.

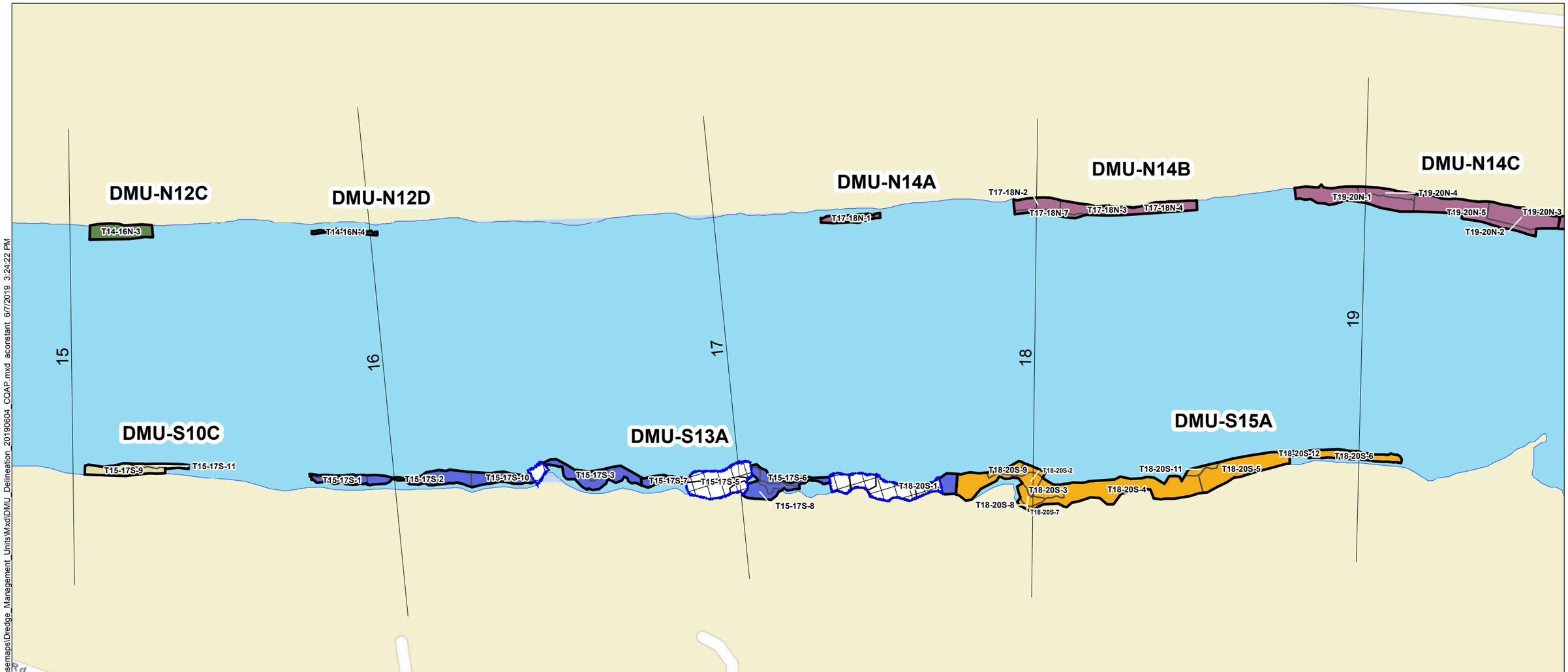
DMU = Dredge Management Unit

Feet
0 100 200

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:24:11 PM

ARCONIC

Figure CA-5
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N12
- DMU-N14
- DMU-S10
- DMU-S13
- DMU-S15

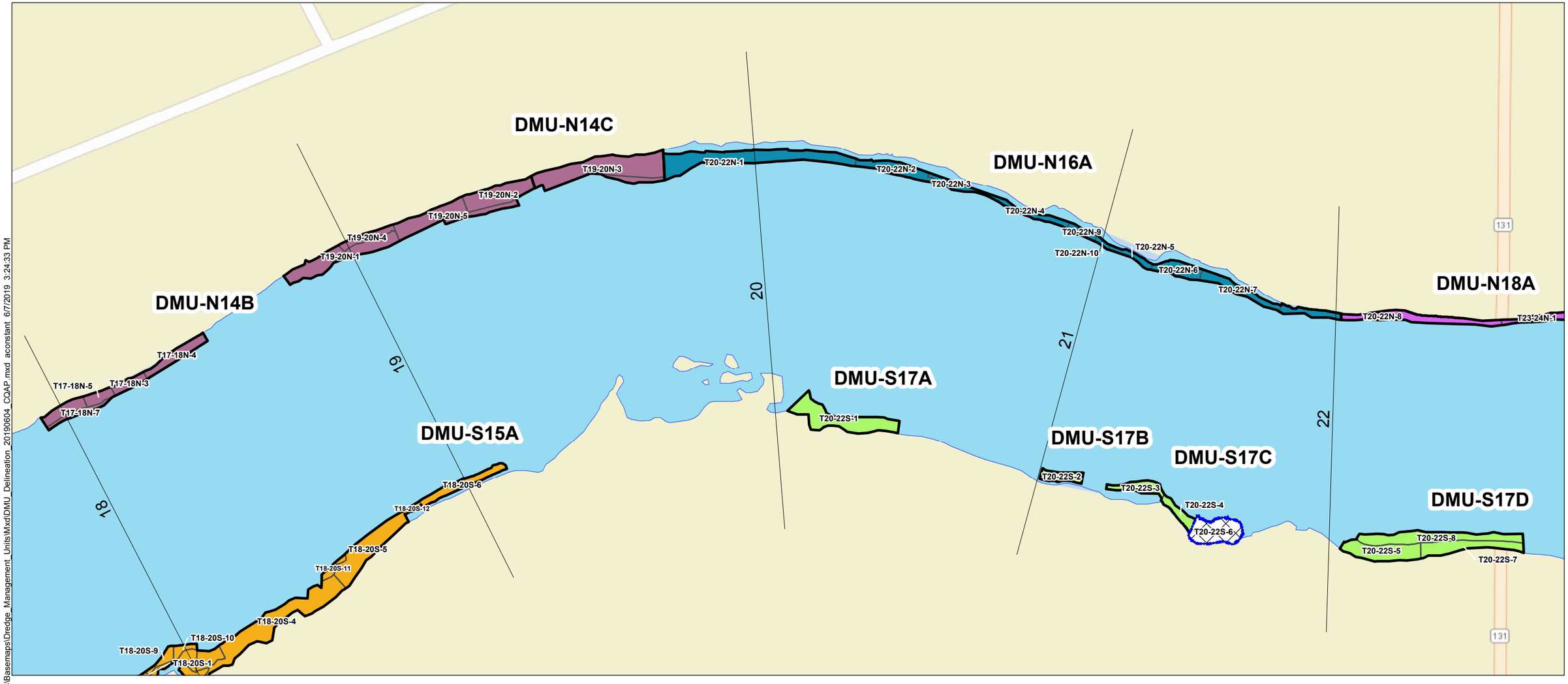
NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:24:22 PM

ARCONIC

Figure CA-6
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N14
- DMU-N16
- DMU-N18
- DMU-S15
- DMU-S17

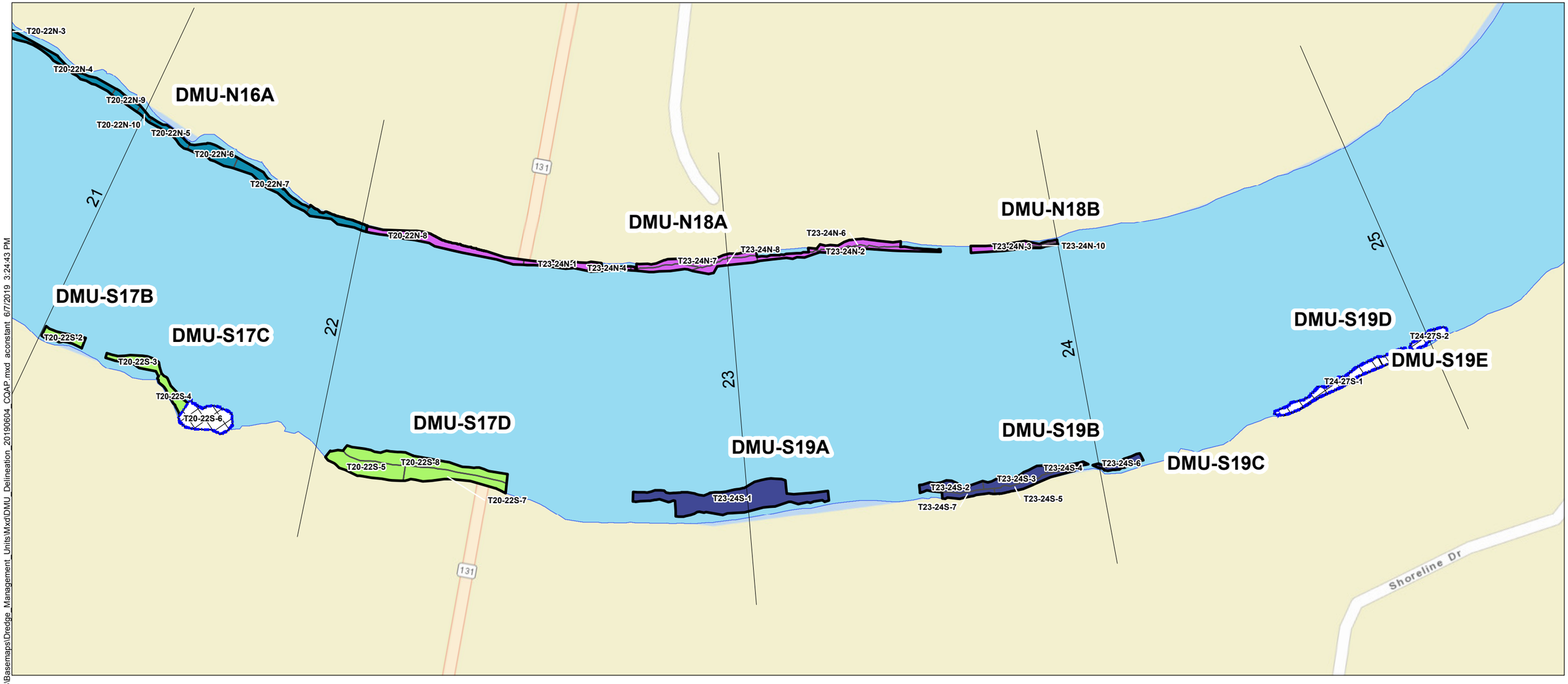
NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:24:33 PM

ARCONIC

Figure CA-7
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N16
- DMU-N18
- DMU-S17
- DMU-S19

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.

DMU = Dredge Management Unit

Feet
0 100 200

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ARCONIC

Figure CA-8
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic

Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N18
- DMU-N20
- DMU-S19
- DMU-S21

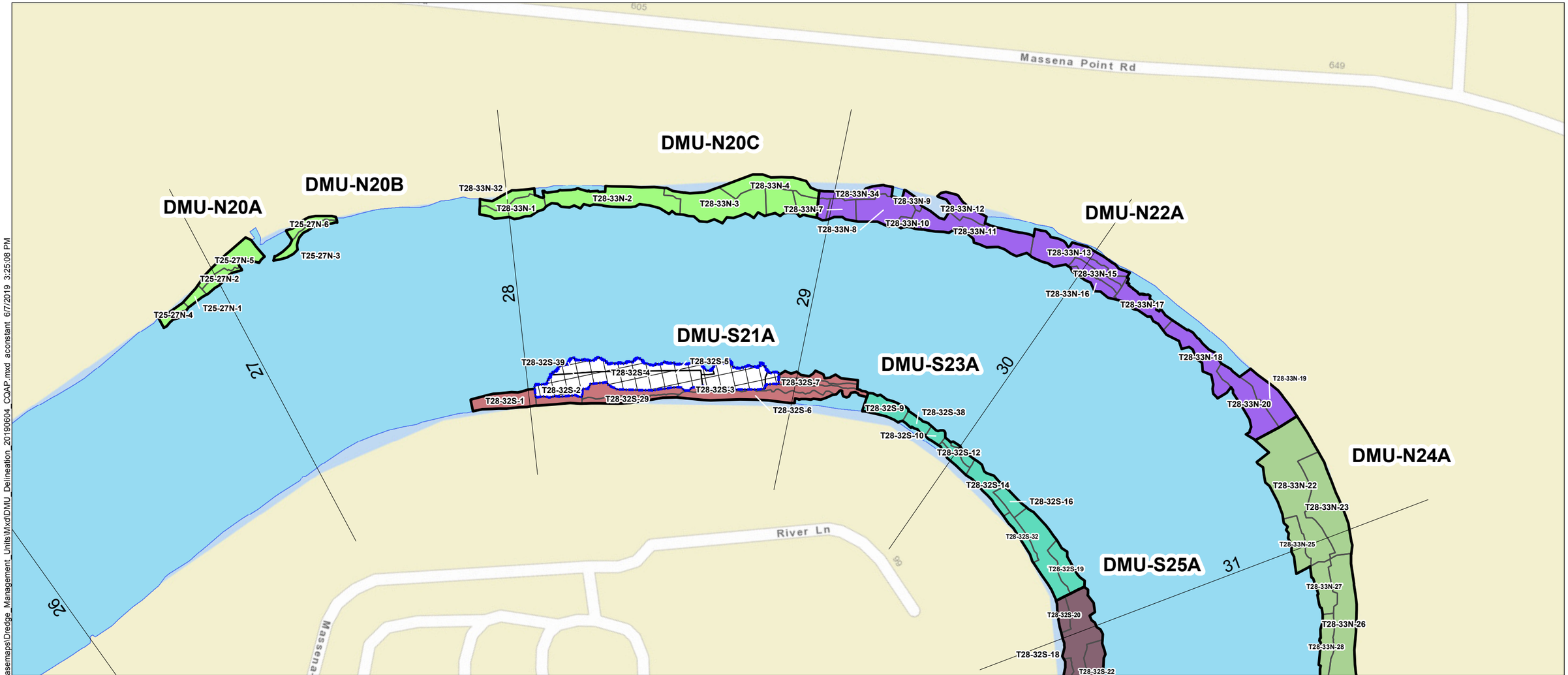
NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:24:56 PM

ARCONIC

Figure CA-9
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N20
- DMU-N22
- DMU-N24
- DMU-S21
- DMU-S23
- DMU-S25

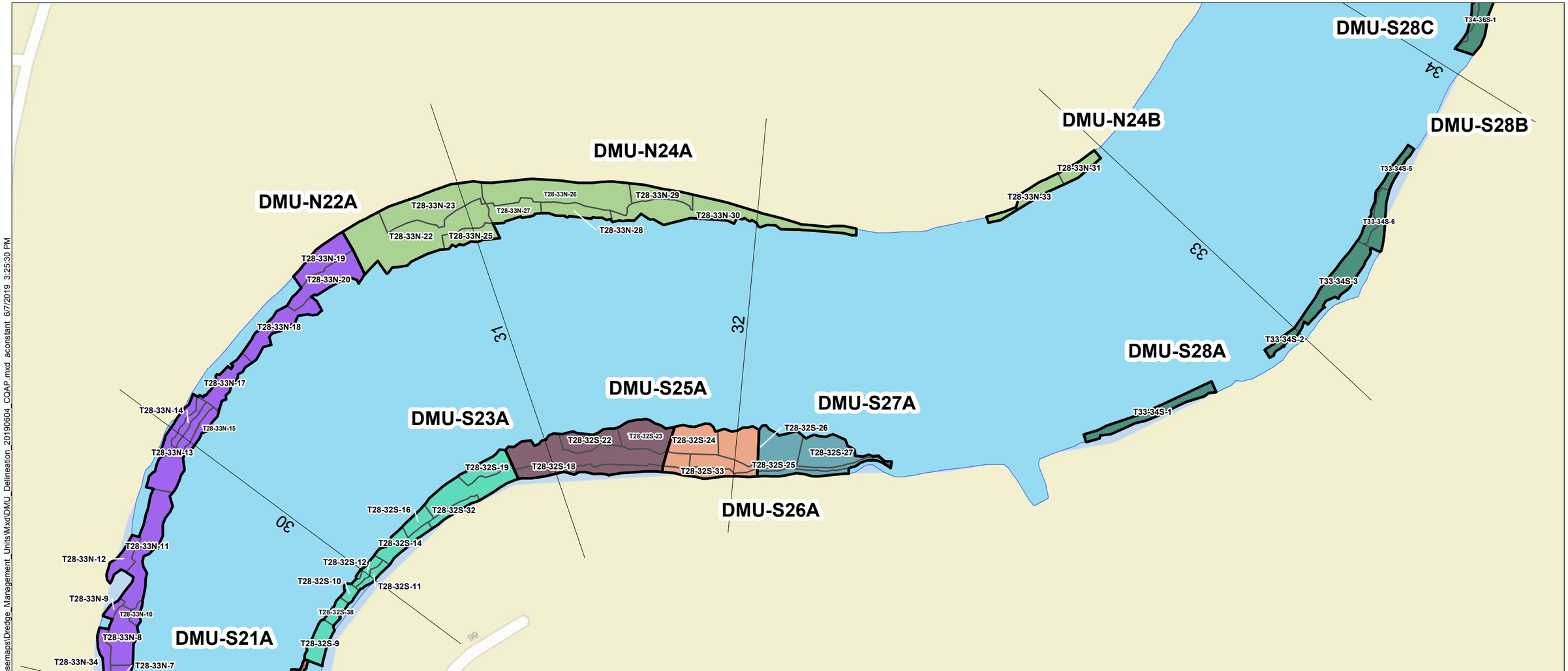
NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:25:08 PM

ARCONIC

Figure CA-10
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N22
- DMU-N24
- DMU-S21
- DMU-S23
- DMU-S25
- DMU-S26
- DMU-S27
- DMU-S28

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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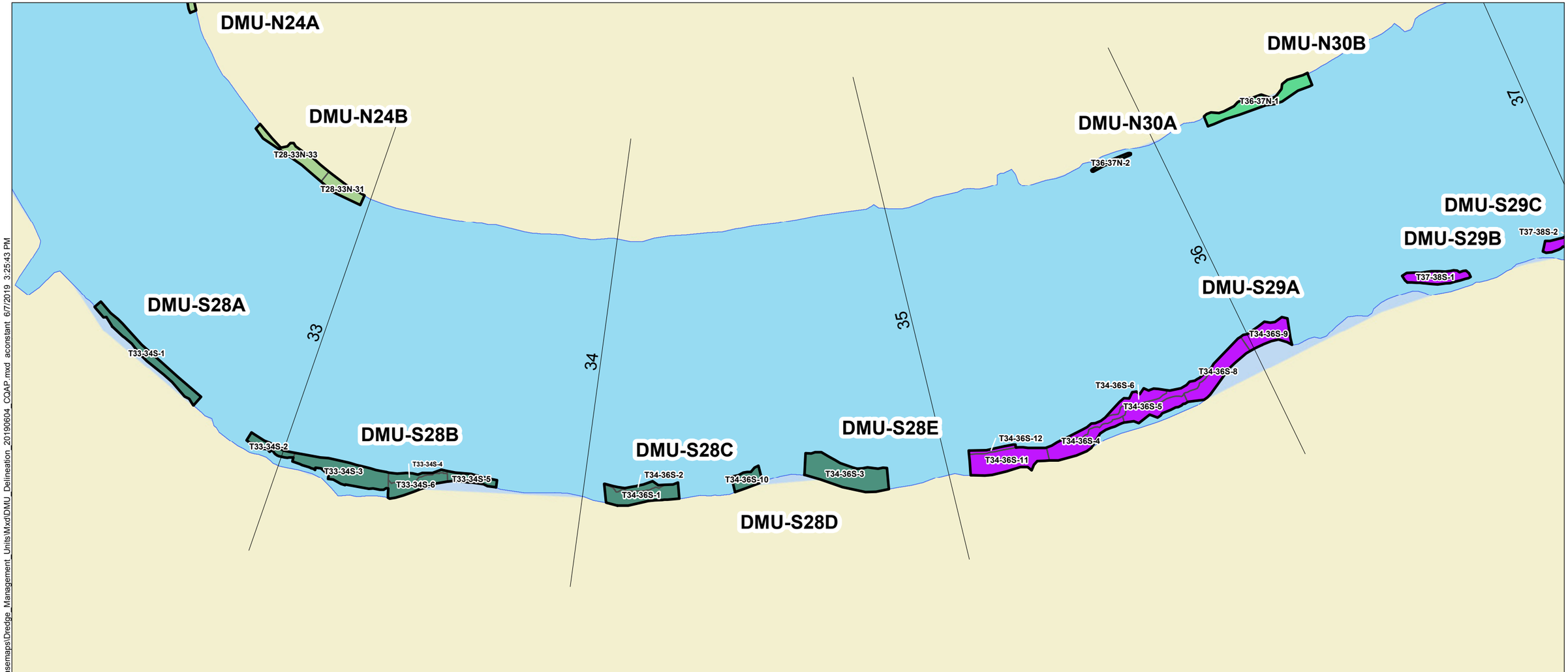
ARCONIC

Figure CA-11
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N24
- DMU-N30
- DMU-S28
- DMU-S29

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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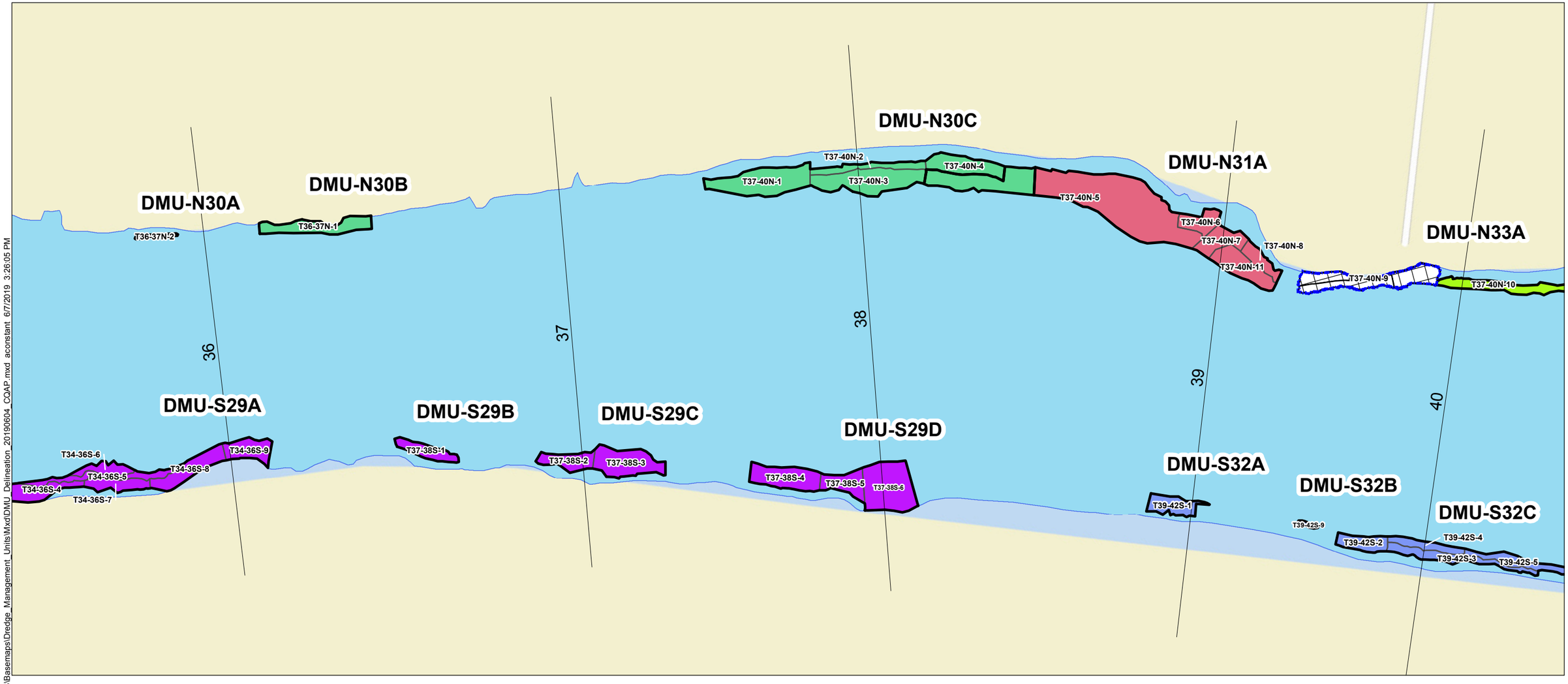
ARCONIC

Figure CA-12
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N30
- DMU-N31
- DMU-N33
- DMU-S29
- DMU-S32

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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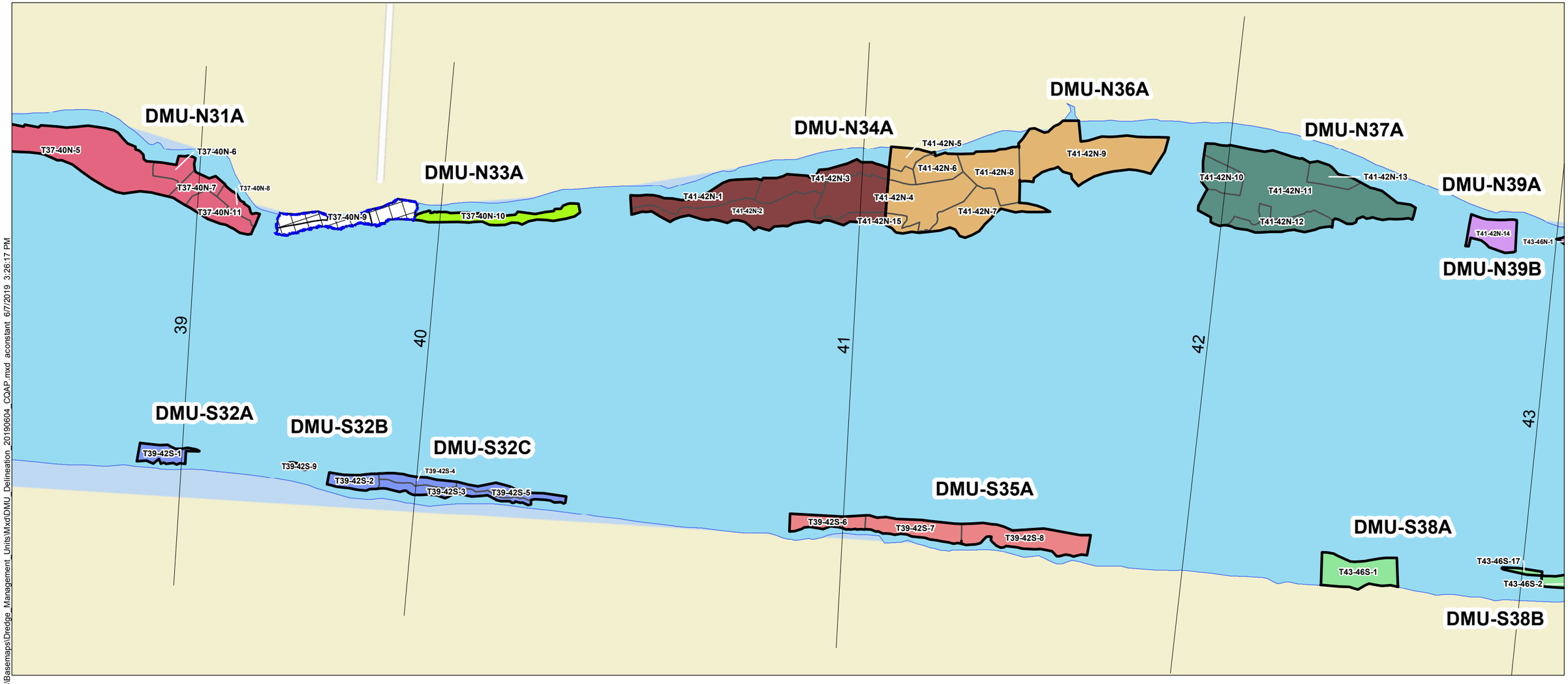
ARCONIC

Figure CA-13
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N31
- DMU-N33
- DMU-N34
- DMU-N36
- DMU-N37
- DMU-N39
- DMU-S32
- DMU-S35
- DMU-S38

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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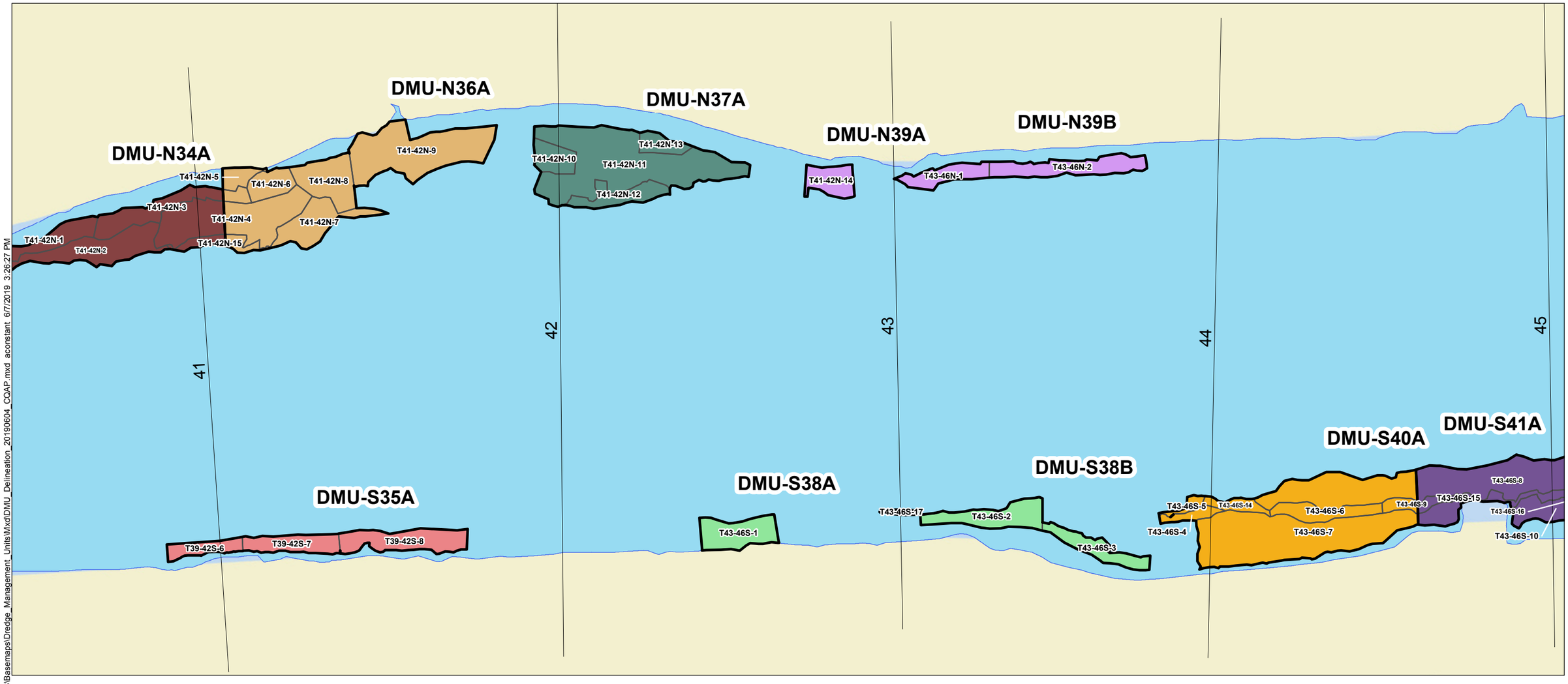
ARCONIC

Figure CA-14
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N34
- DMU-N36
- DMU-N37
- DMU-N39
- DMU-S35
- DMU-S38
- DMU-S40
- DMU-S41

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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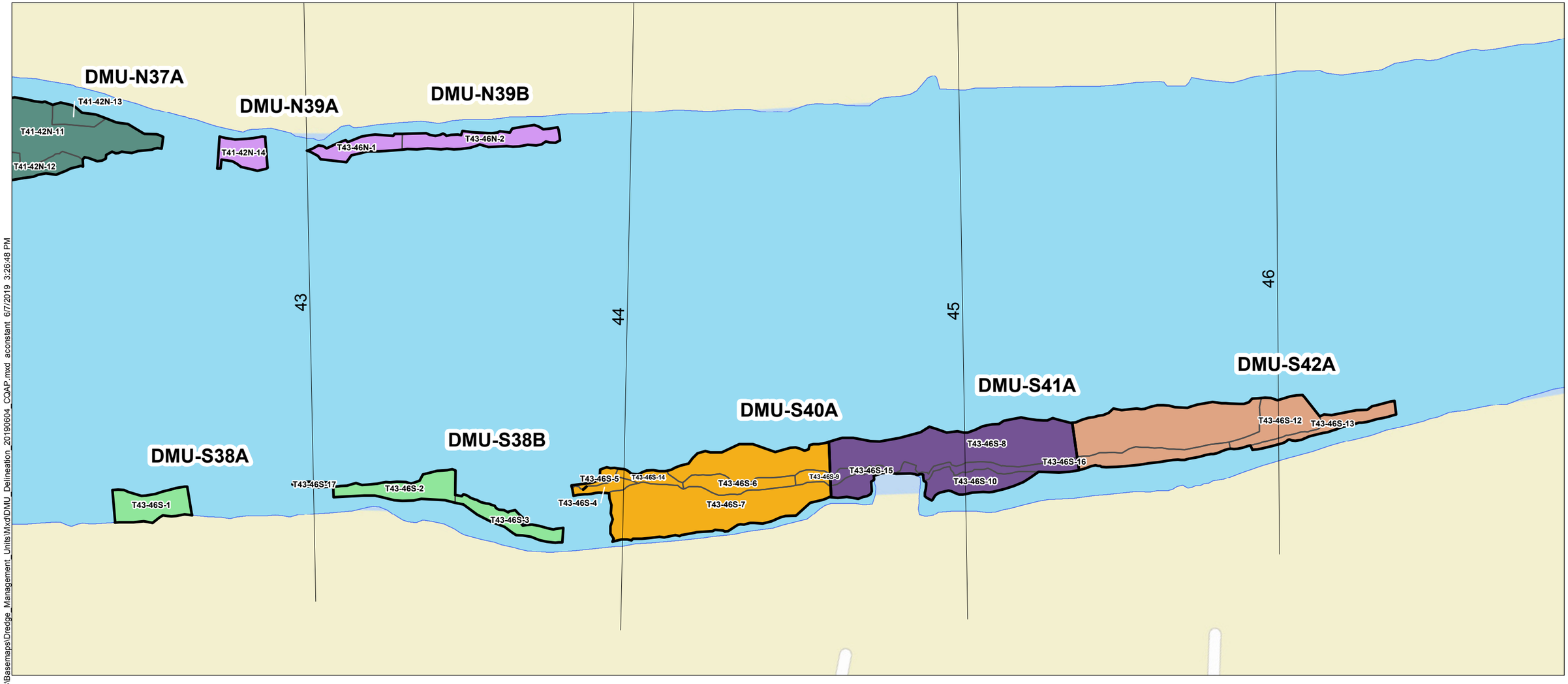
ARCONIC

Figure CA-15
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N37
- DMU-N39
- DMU-S38
- DMU-S40
- DMU-S41
- DMU-S42

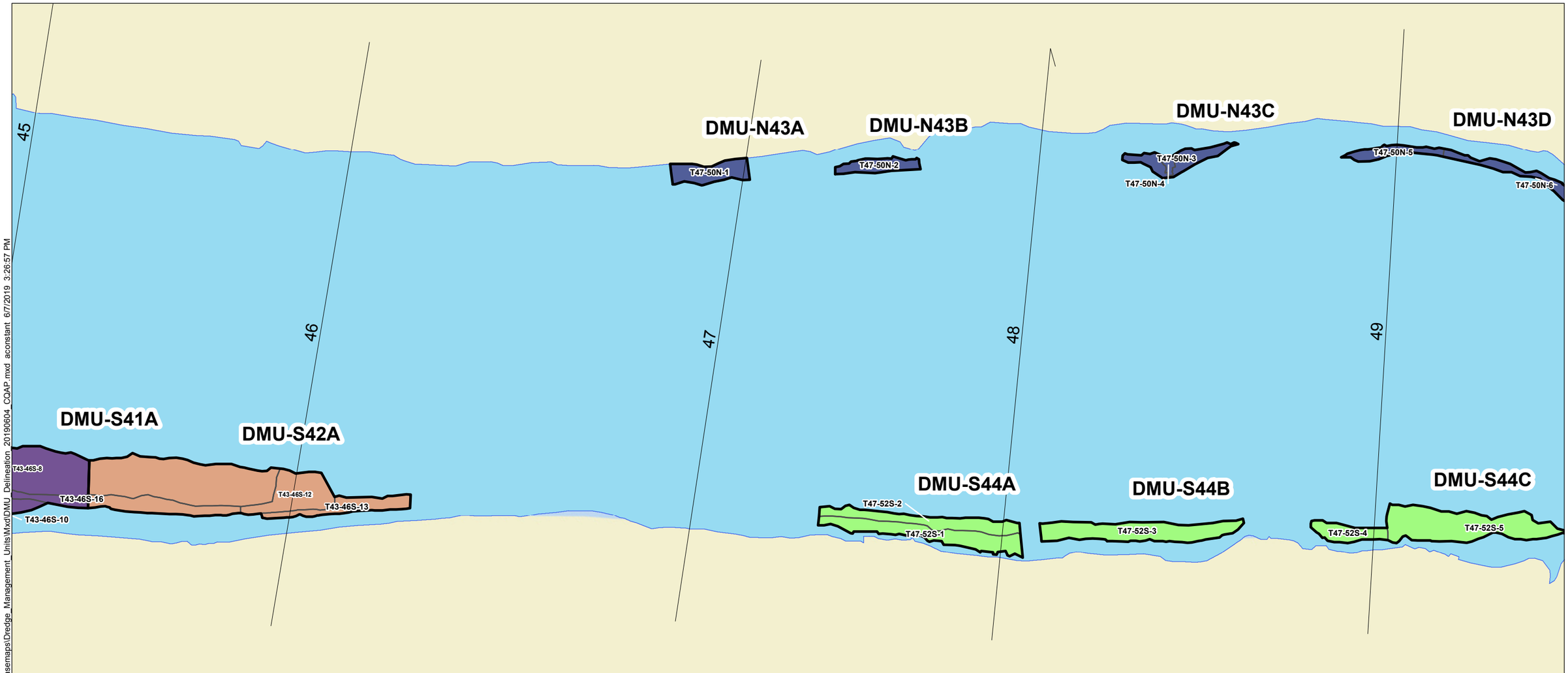
NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:26:48 PM

ARCONIC

Figure CA-16
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N43
- DMU-S41
- DMU-S42
- DMU-S44

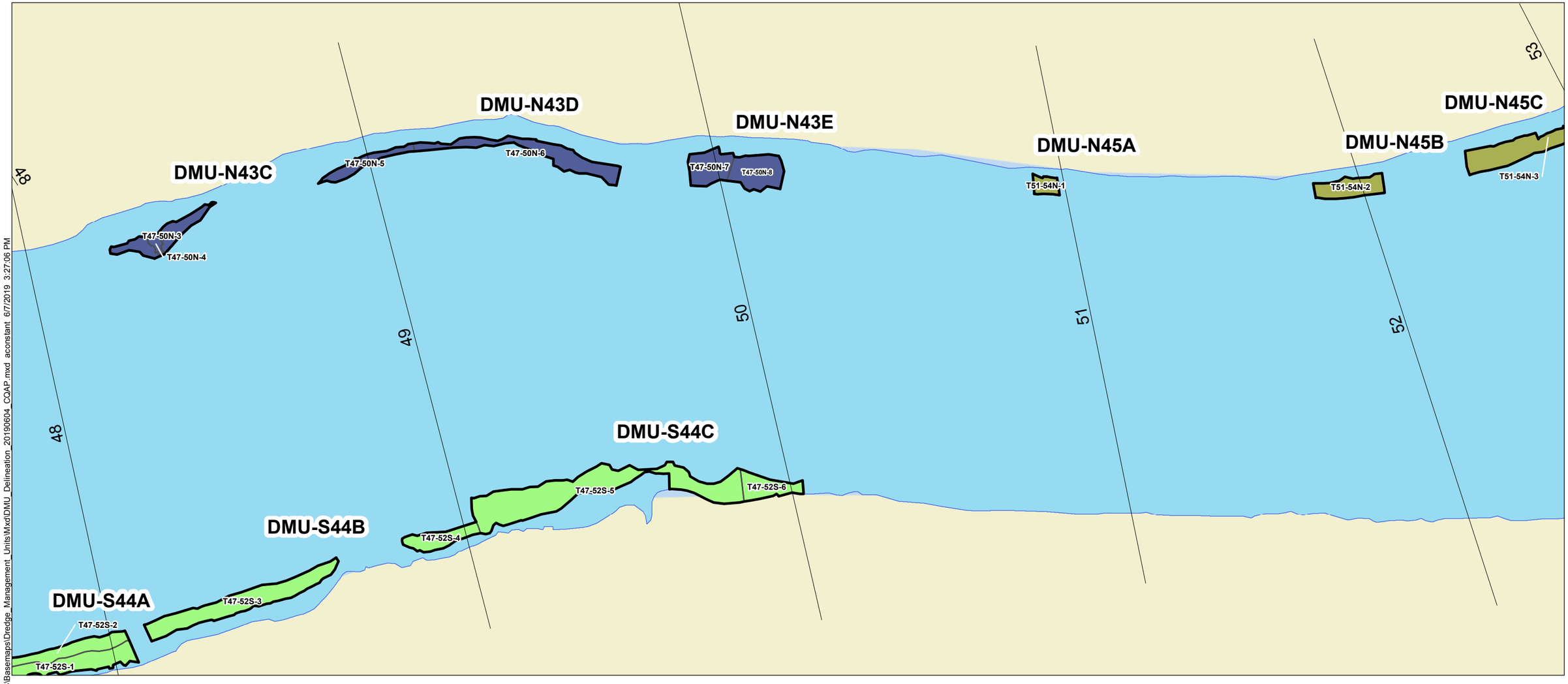
NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:26:57 PM

ARCONIC

Figure CA-17
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N43
- DMU-N45
- DMU-S44

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.

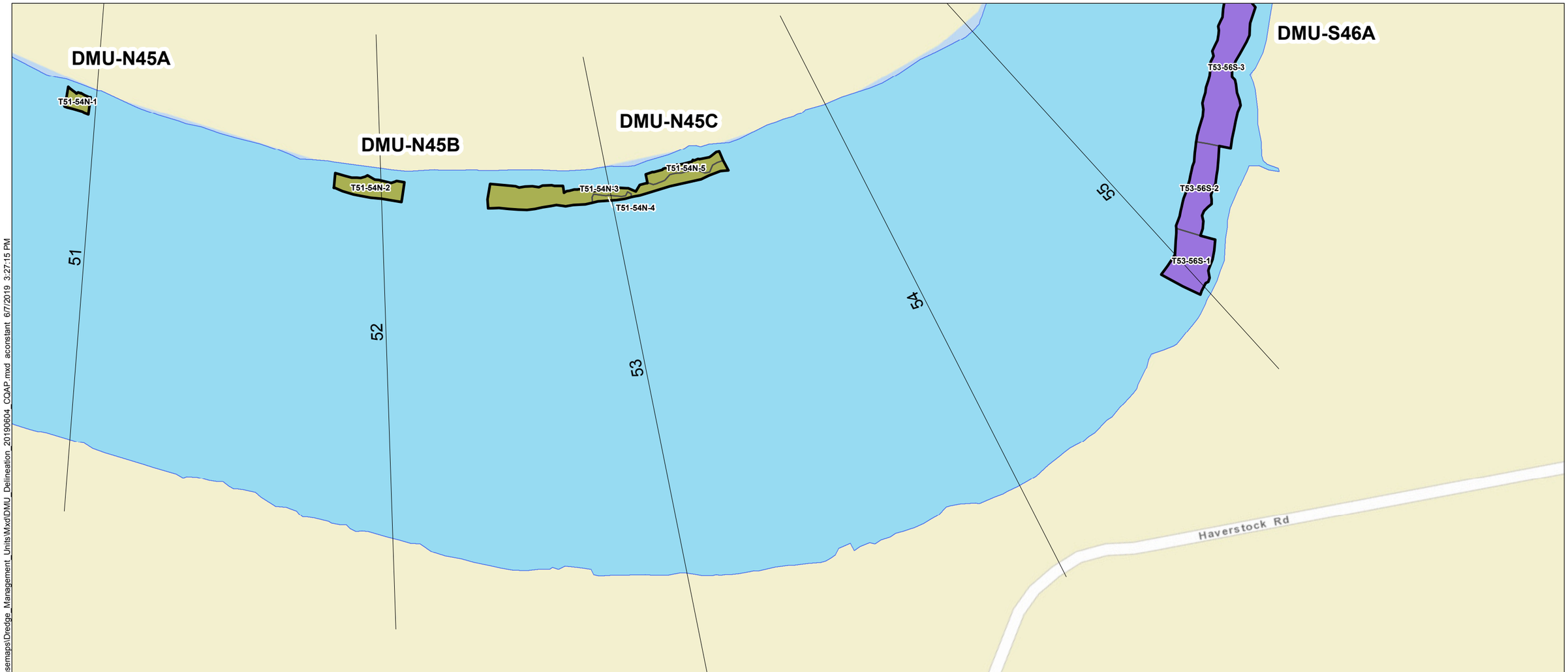
DMU = Dredge Management Unit

Feet
0 100 200

\\saratoga1\saratoga\Projects\Alcoa\Grasse_River\GIS\Basemaps\Dredge_Management_Units\Mxd\DMU_Delineation_20190604_CQAP.mxd aconstant 6/7/2019 3:27:06 PM

ARCONIC

Figure CA-18
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N45
- DMU-S46

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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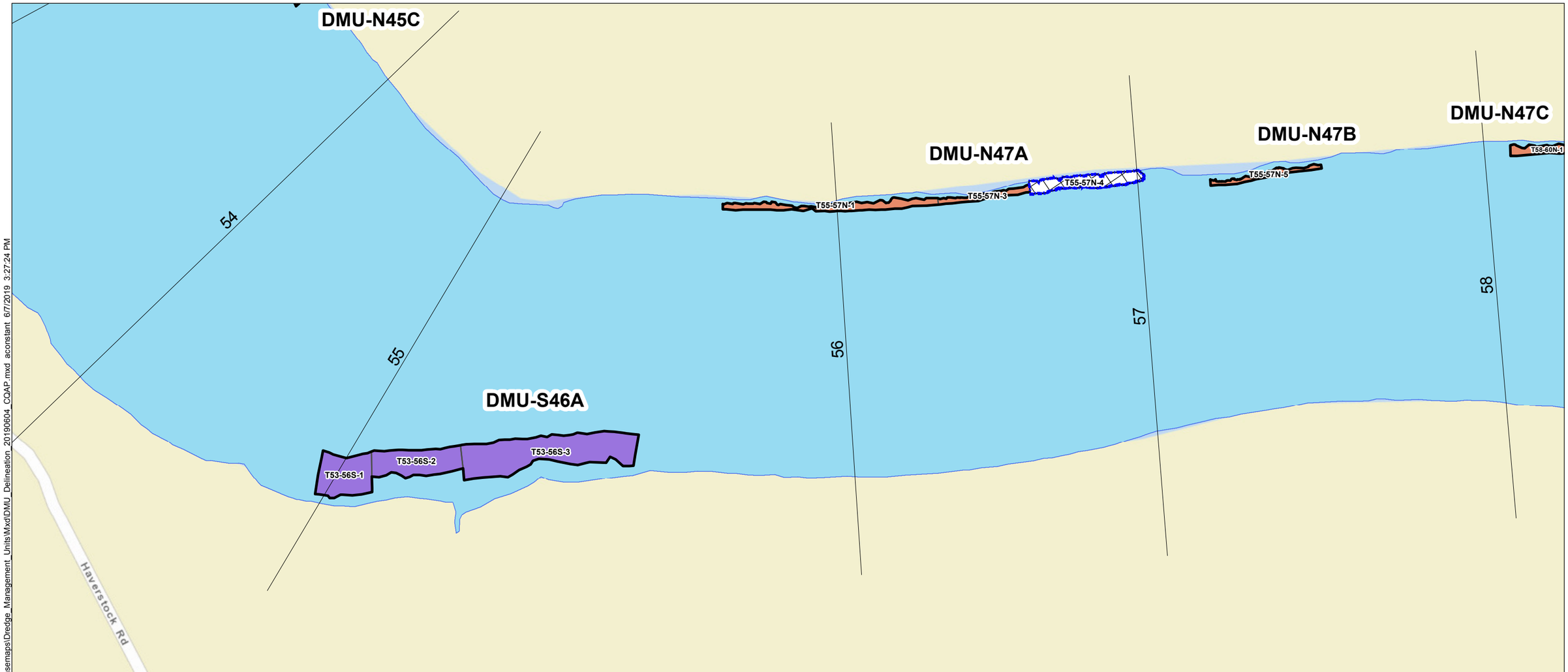
ARCONIC

Figure CA-19
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N45
- DMU-N47
- DMU-S46

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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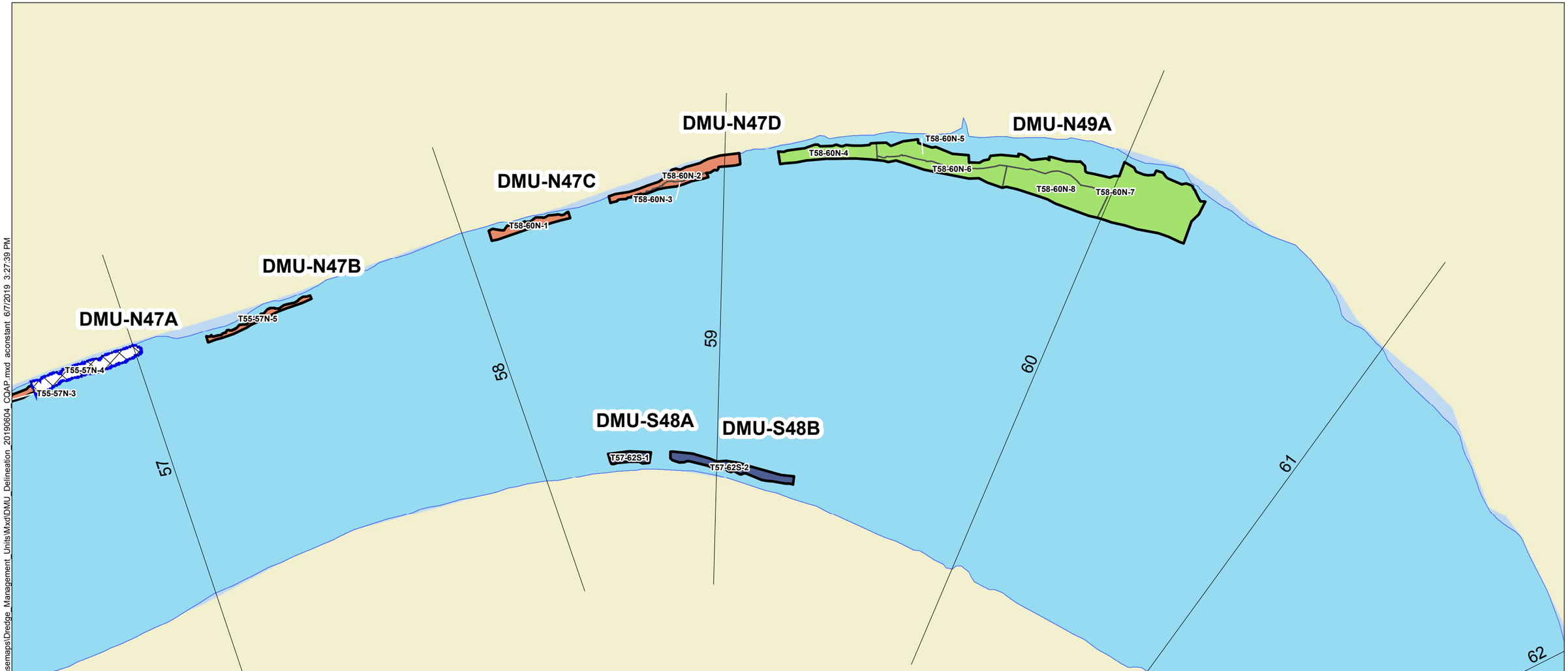
ARCONIC

Figure CA-20
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N47
- DMU-N49
- DMU-S48

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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ARCONIC

Figure CA-21
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic

Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N49
- DMU-N50
- DMU-S51

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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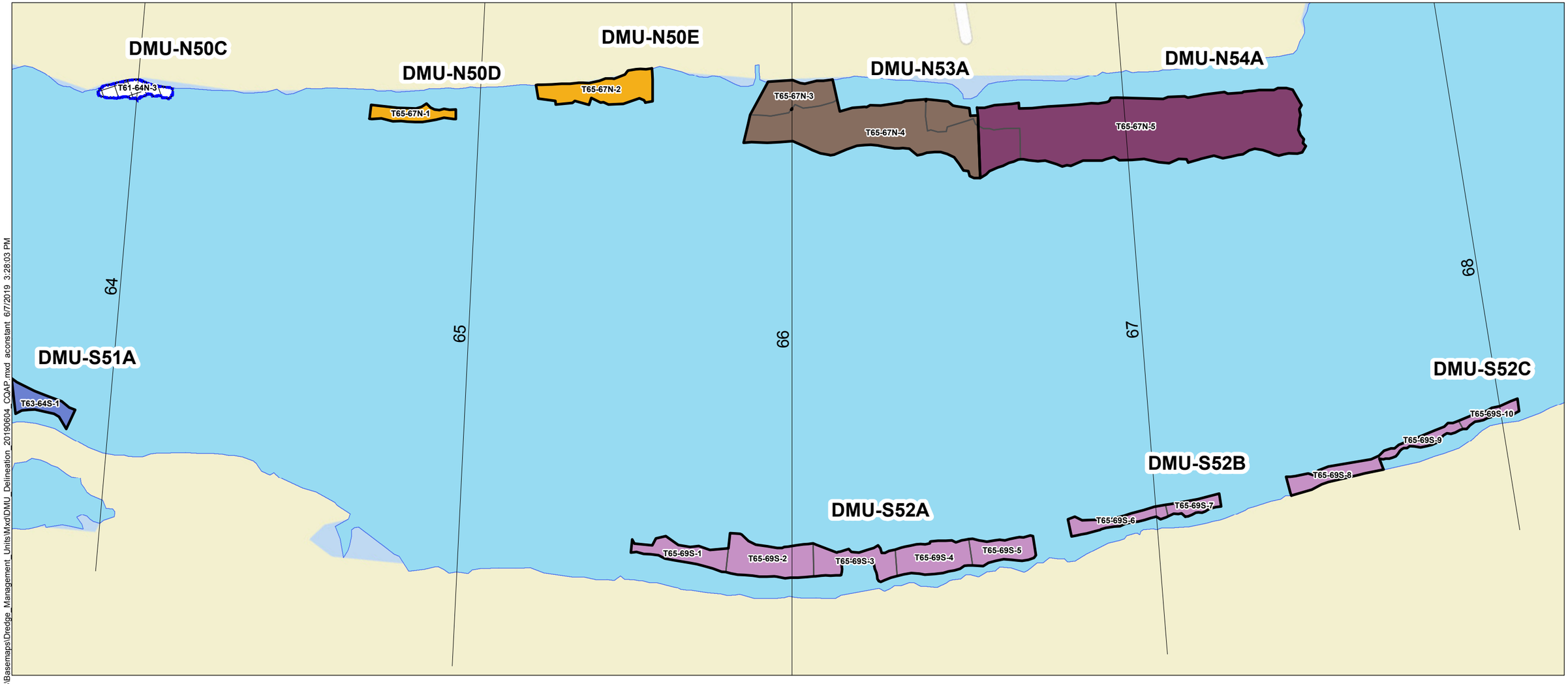
ARCONIC

Figure CA-22
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N50
- DMU-N53
- DMU-N54
- DMU-S51
- DMU-S52

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.

DMU = Dredge Management Unit

Feet
0 100 200

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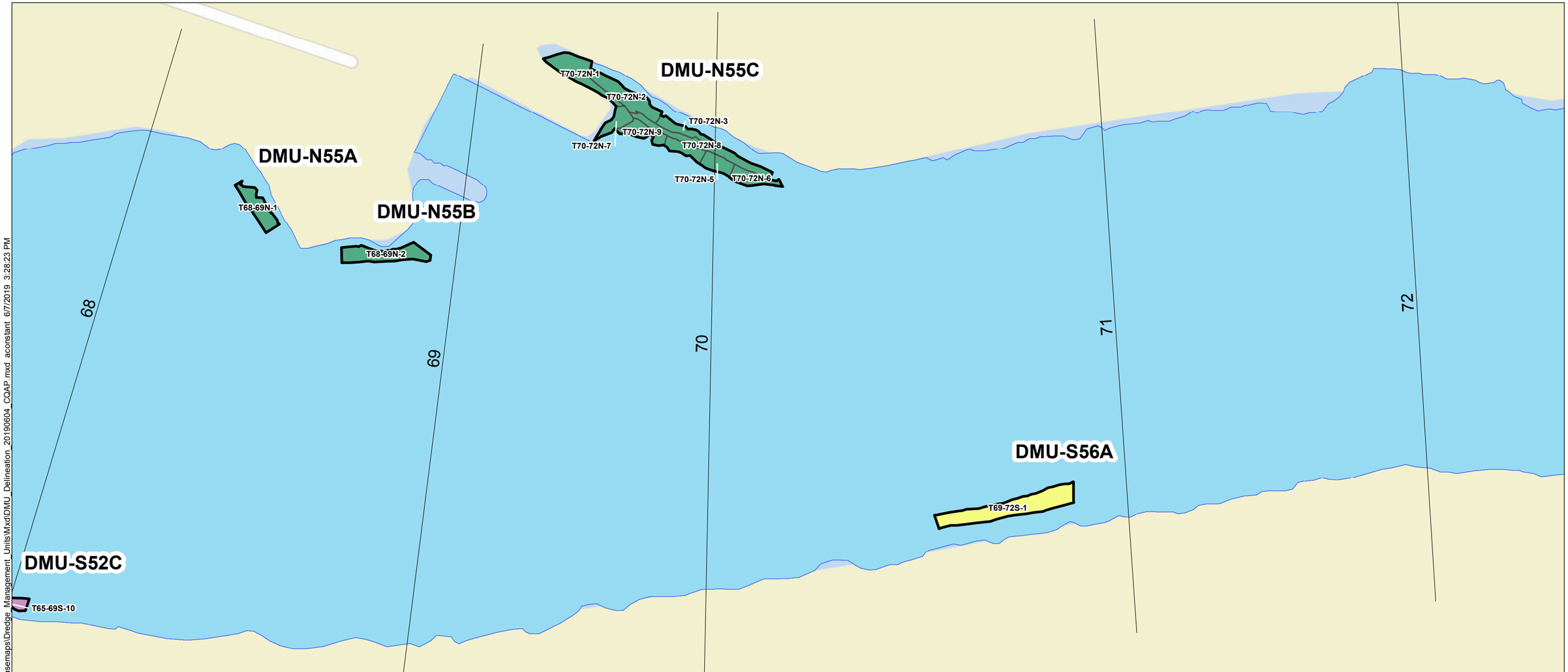
Figure CA-23
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic



Legend

- Design Dredge Limits
- Dredge Area and Subarea Label
- Interval Dredge Area
- Grasse River
- Grasse River Transects

***Color shading denotes DMU area**

- DMU-N55
- DMU-S52
- DMU-S56

NOTE:
1. Remedial areas based on 2019 Final Design Report (FDR).
2. Proposed DMUs prepared by Anchor QEA and Brennan.
DMU = Dredge Management Unit

Feet
0 100 200

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Figure CA-24
Dredge Management Unit Delineation
Construction Quality Assurance Plan
Grasse River Project/Arconic

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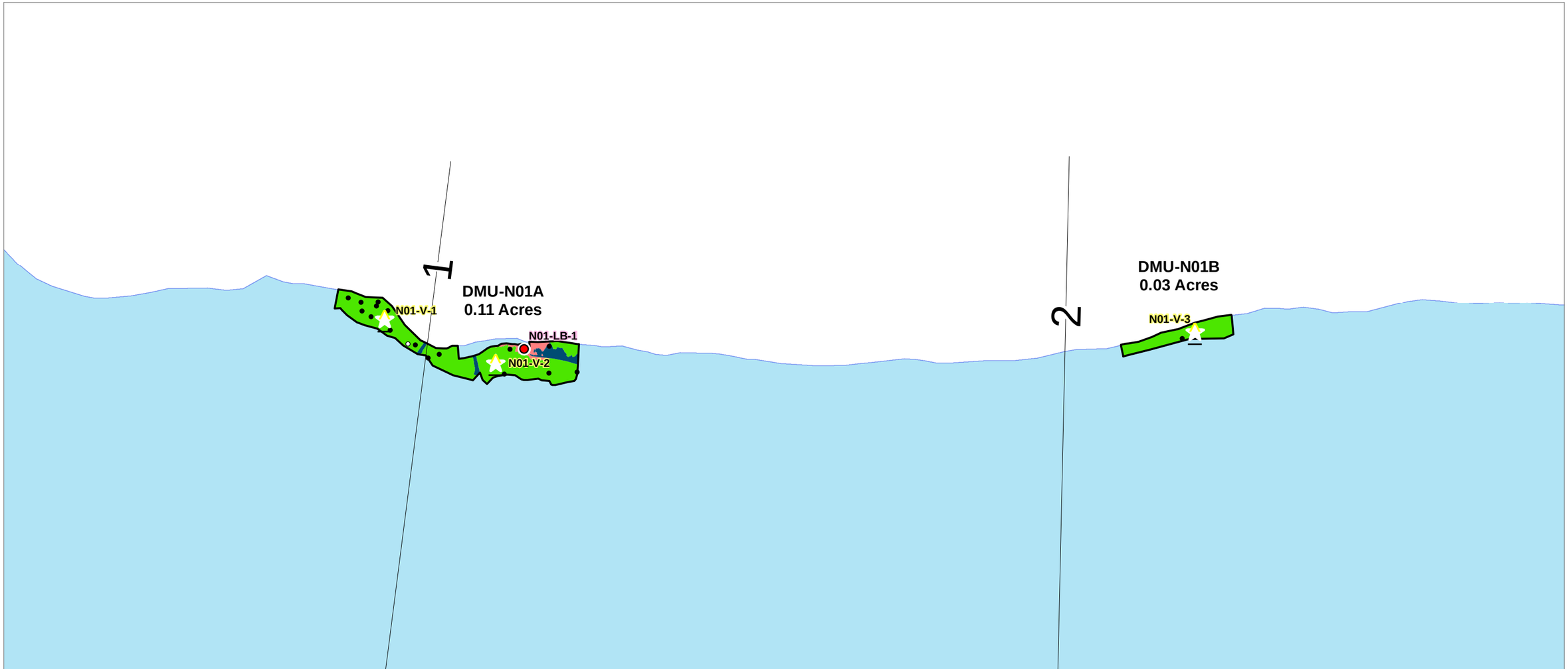

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N01 are shown.
DMU = Dredge Management Unit

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Figure CB-1
Target Post-Dredge Sampling Locations: DMU-N01
Construction Quality Assurance Plan
Grasse River Project/Arconic

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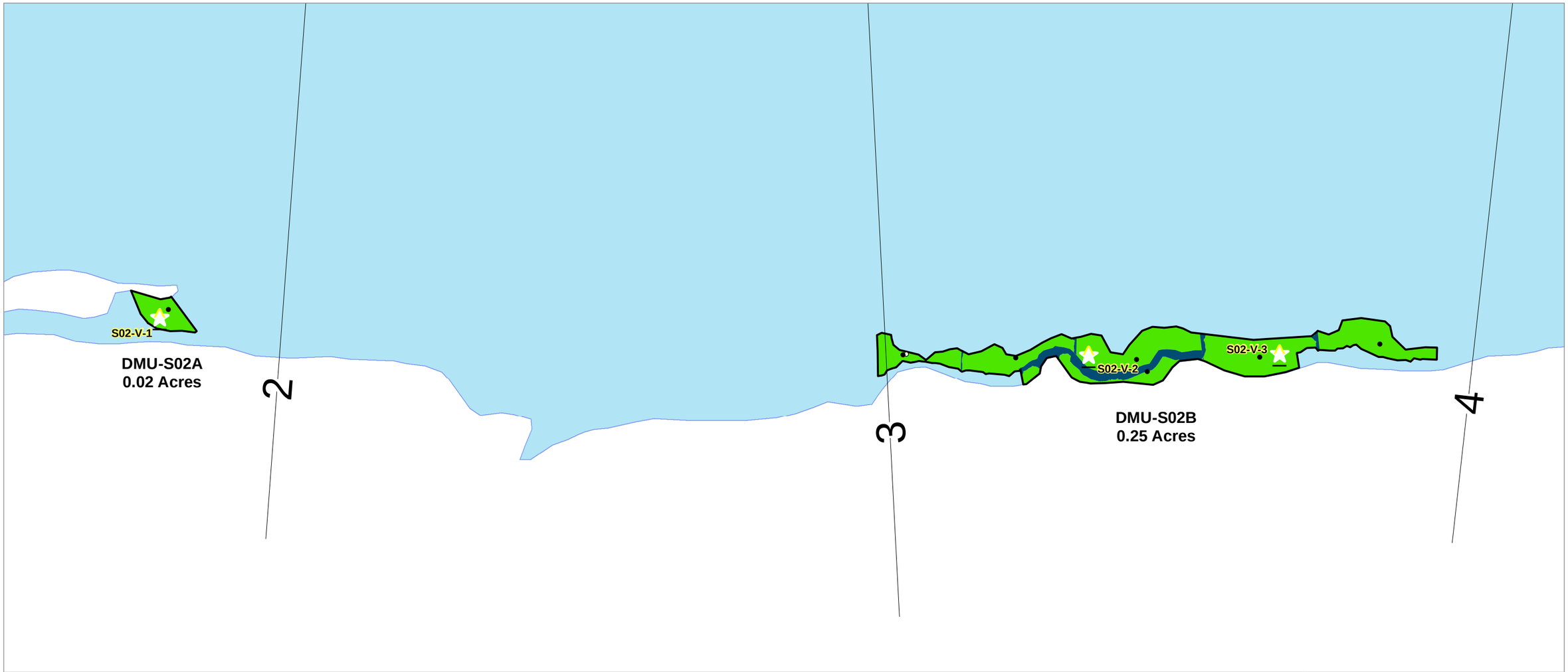

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S02 are shown.
DMU = Dredge Management Unit

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Feet

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ARCONIC

Figure CB-2
Target Post-Dredge Sampling Locations: DMU-S02

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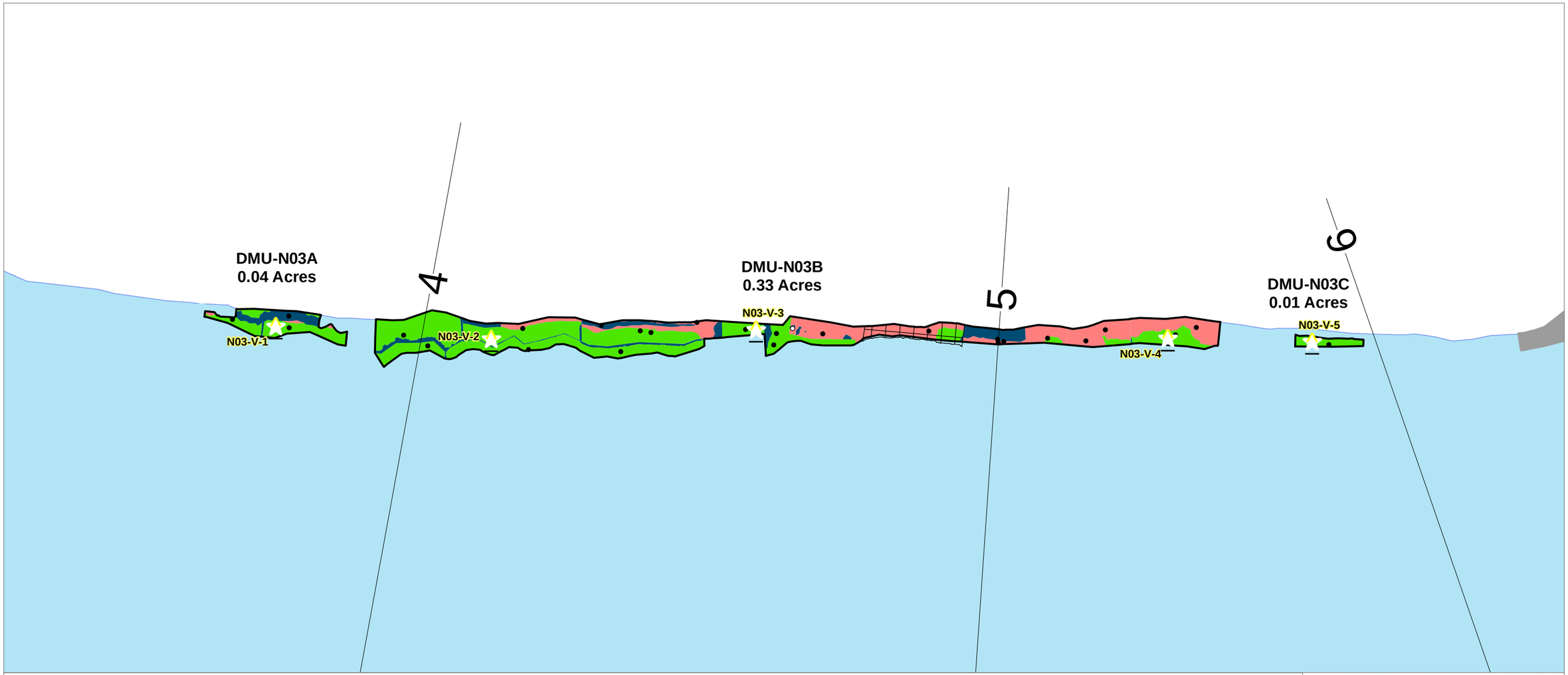

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N03 are shown.
DMU = Dredge Management Unit

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Feet

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Figure CB-3
Target Post-Dredge Sampling Locations: DMU-N03
Construction Quality Assurance Plan
Grasse River Project/Arconic

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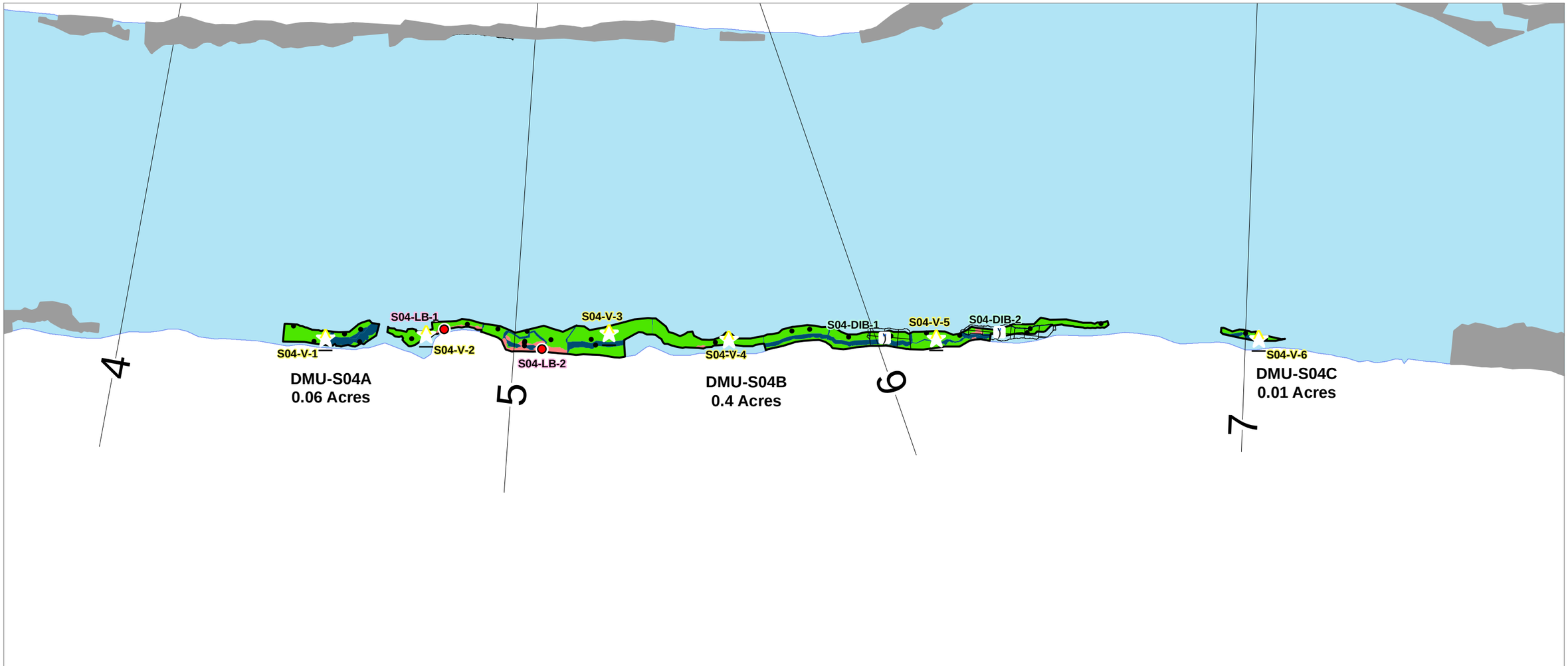

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S04 are shown.
DMU = Dredge Management Unit

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Feet

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ARCONIC

Figure CB-4
Target Post-Dredge Sampling Locations: DMU-S04
Construction Quality Assurance Plan
Grasse River Project/Arconic

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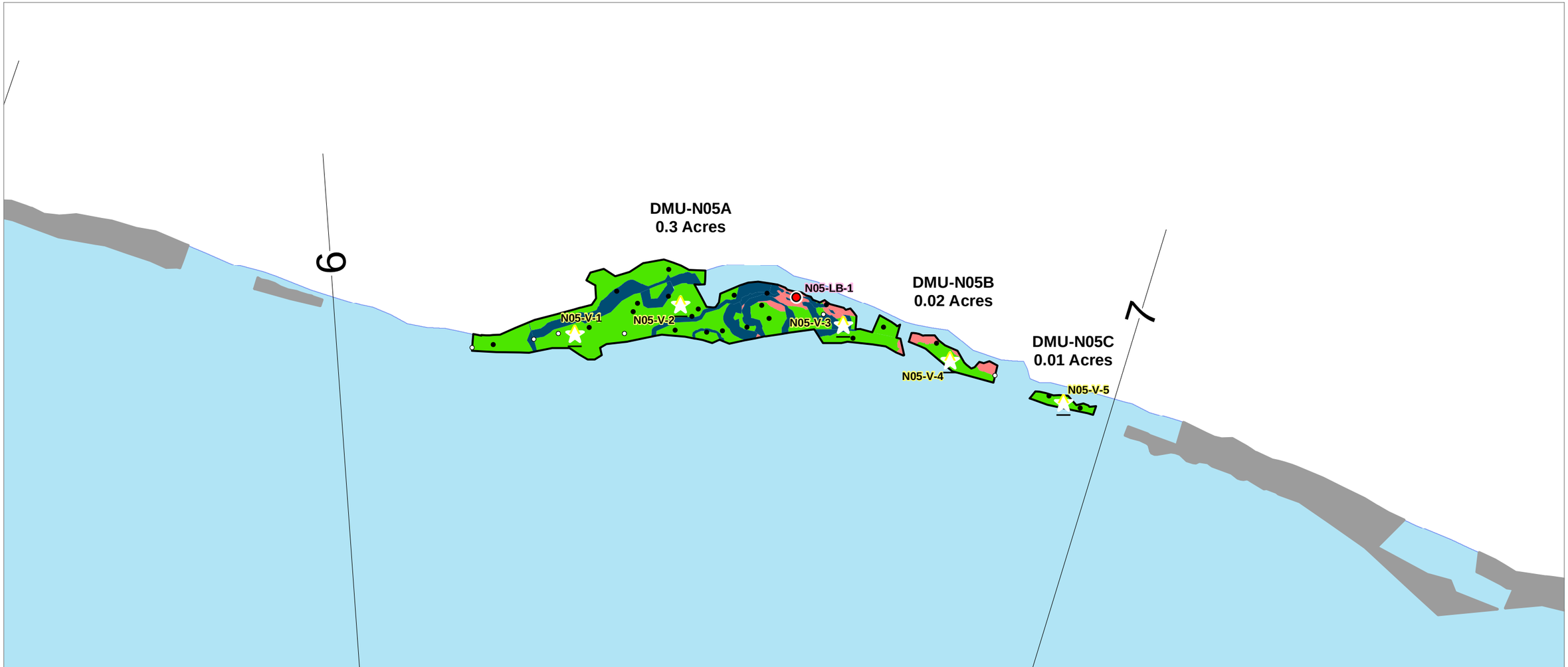

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N05 are shown.
DMU = Dredge Management Unit

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ARCONIC

Figure CB-5
Target Post-Dredge Sampling Locations: DMU-N05

Construction Quality Assurance Plan
Grasse River Project/Arconic

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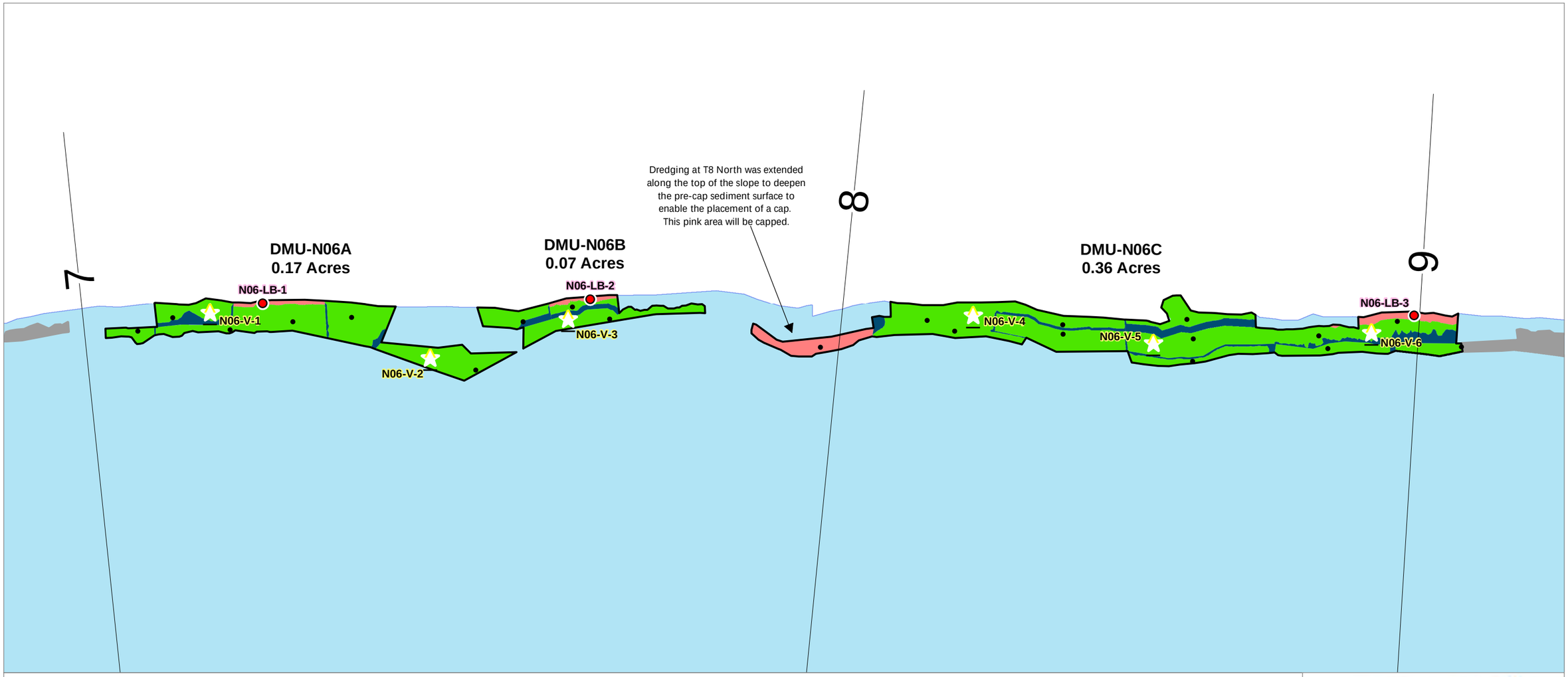

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N06 are shown.
DMU = Dredge Management Unit

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Feet

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ARCONIC

Figure CB-6
Target Post-Dredge Sampling Locations: DMU-N06
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S07 are shown.
DMU = Dredge Management Unit

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Feet

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Figure CB-7
Target Post-Dredge Sampling Locations: DMU-S07

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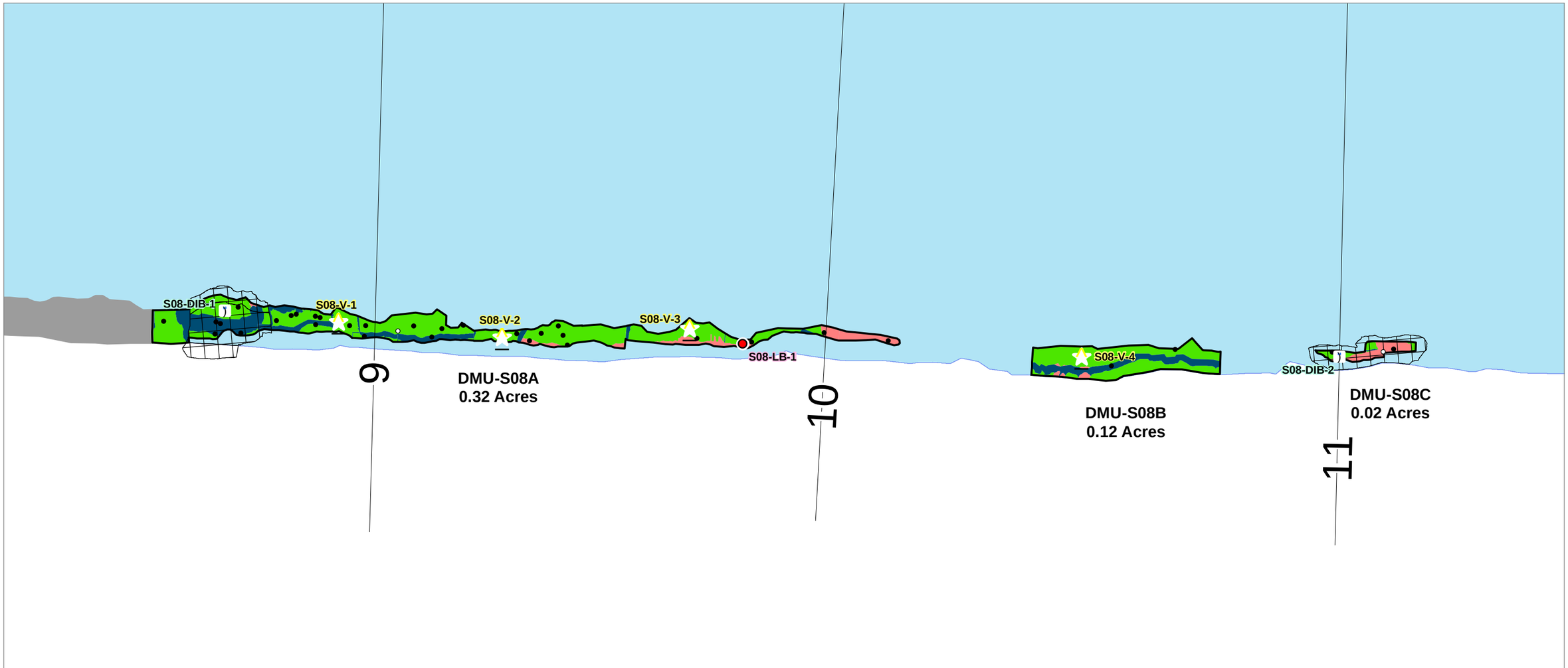

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S08 are shown.
DMU = Dredge Management Unit

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Feet

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Figure CB-8
Target Post-Dredge Sampling Locations: DMU-S08
Construction Quality Assurance Plan
Grasse River Project/Arconic

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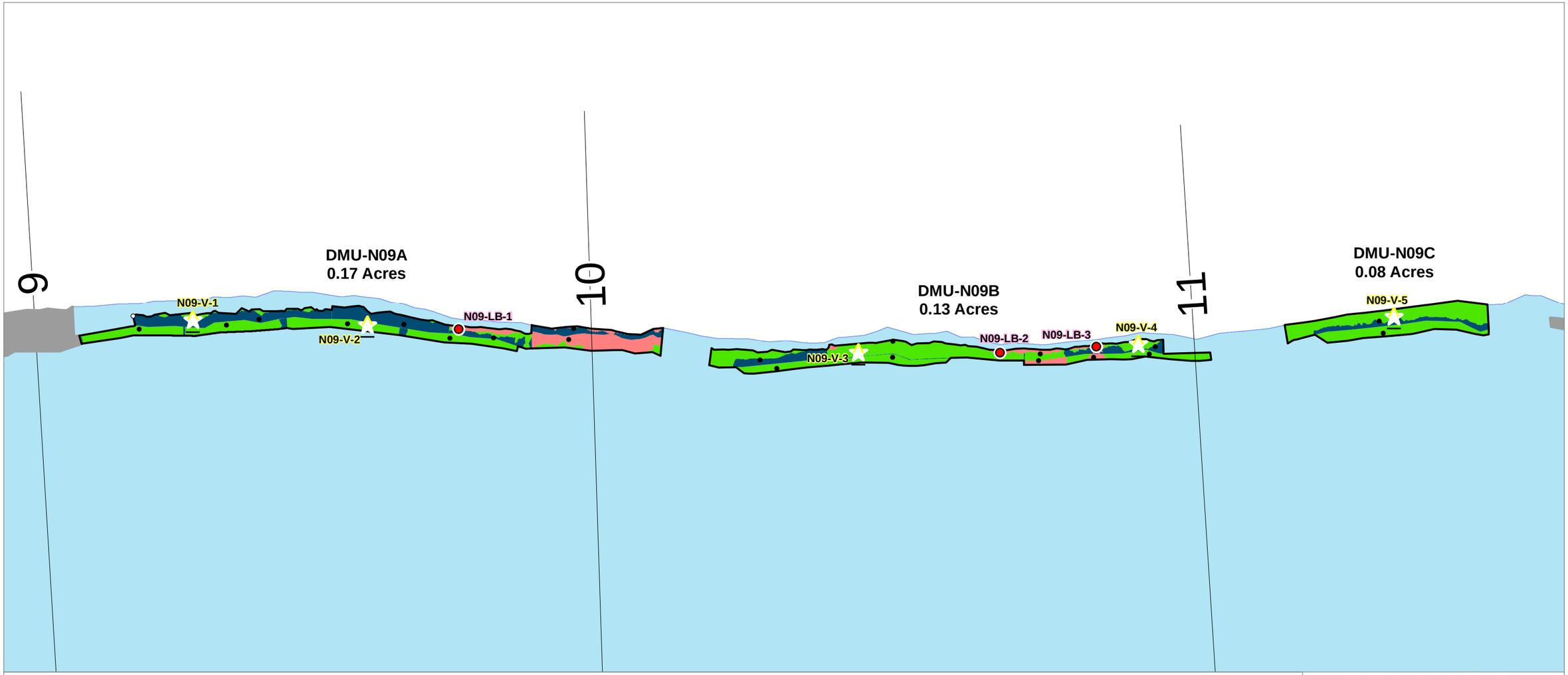

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N09 are shown.
DMU = Dredge Management Unit

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Feet

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ARCONIC

Figure CB-9
Target Post-Dredge Sampling Locations: DMU-N09
Construction Quality Assurance Plan
Grasse River Project/Arconic

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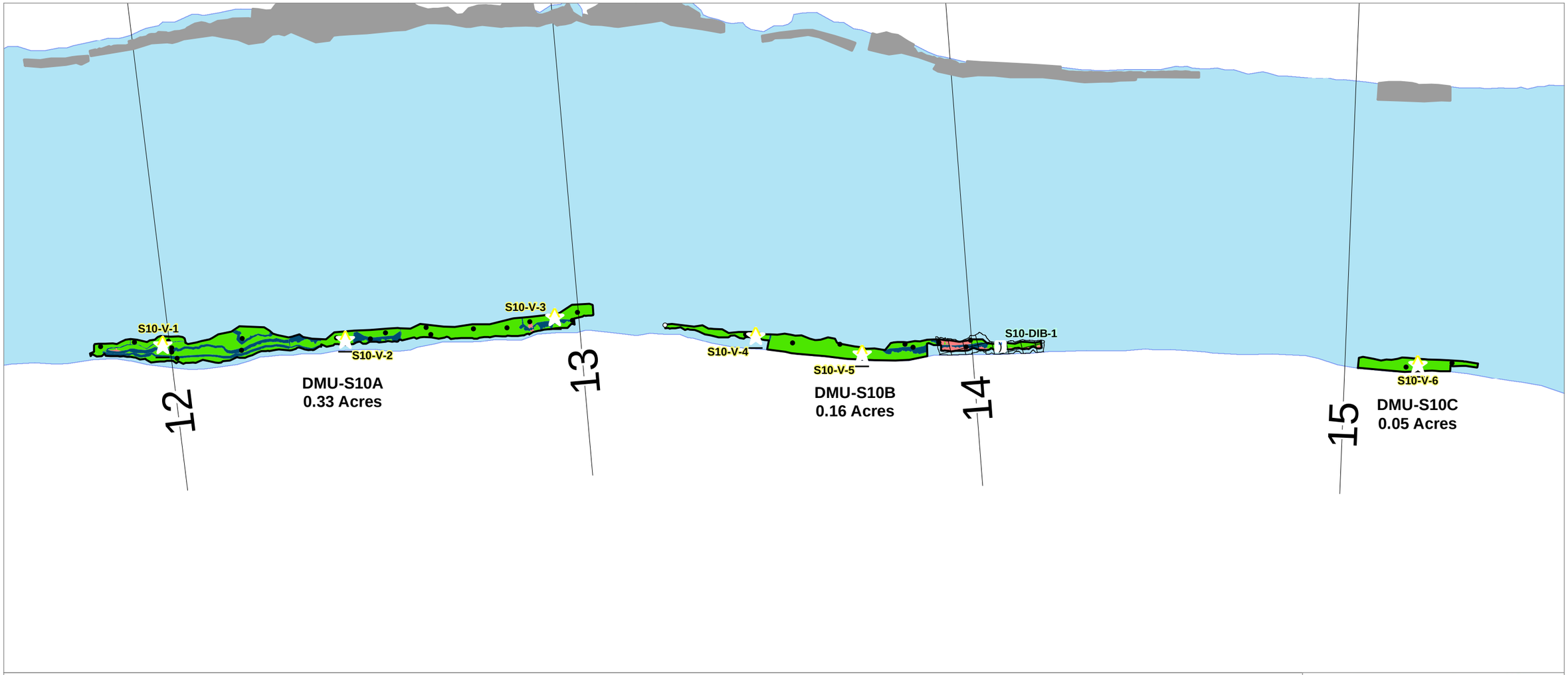

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S10 are shown.
DMU = Dredge Management Unit

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ARCONIC

Figure CB-10
Target Post-Dredge Sampling Locations: DMU-S10
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N11 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-11
Target Post-Dredge Sampling Locations: DMU-N11
Construction Quality Assurance Plan
Grasse River Project/Arconic

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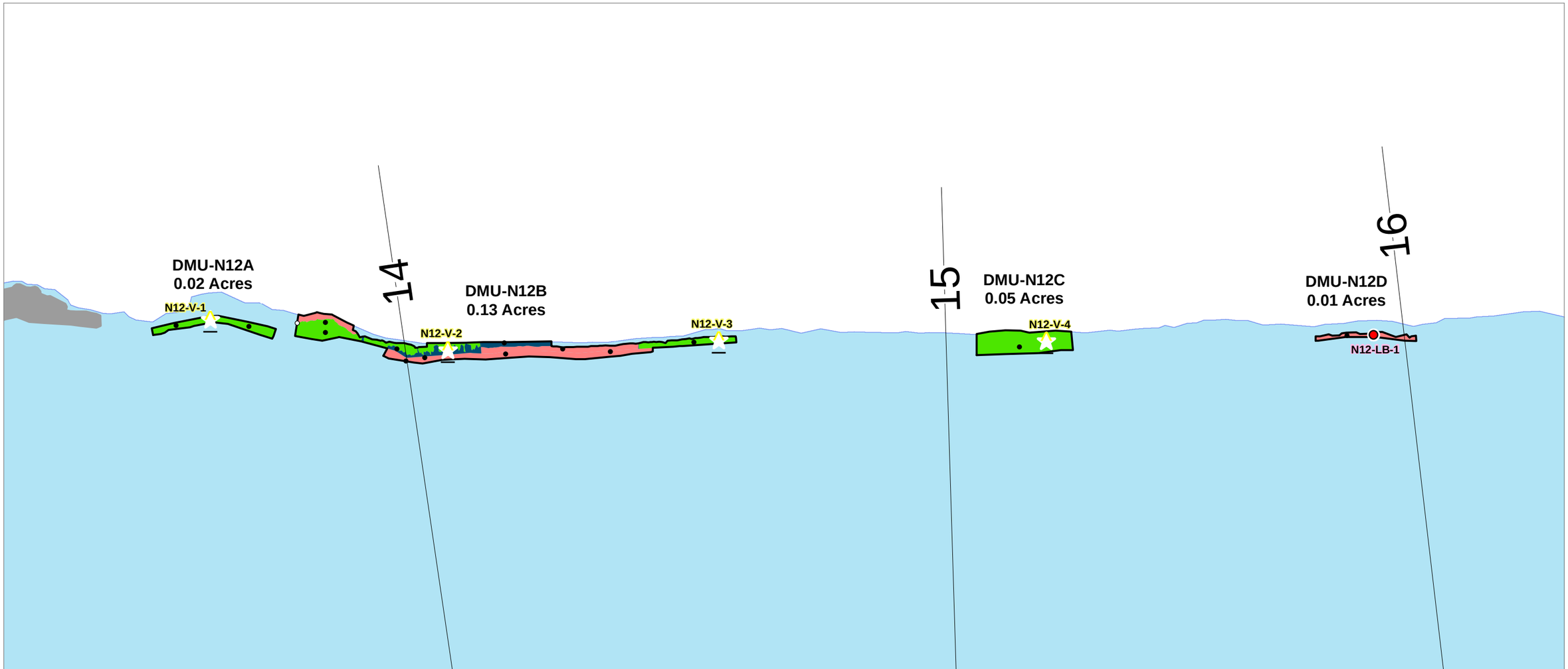

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N12 are shown.
DMU = Dredge Management Unit

0 100
Feet

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ARCONIC

Figure CB-12
Target Post-Dredge Sampling Locations: DMU-N12
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

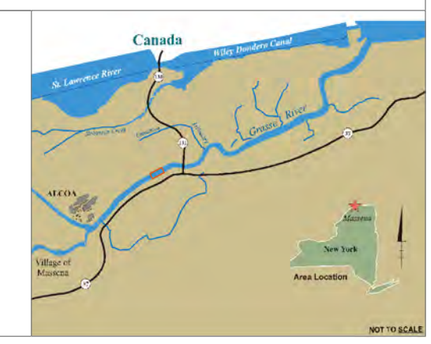
- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S13 are shown.
DMU = Dredge Management Unit

0 80
Feet



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ARCONIC

Figure CB-13
Target Post-Dredge Sampling Locations: DMU-S13
Construction Quality Assurance Plan
Grasse River Project/Arconic

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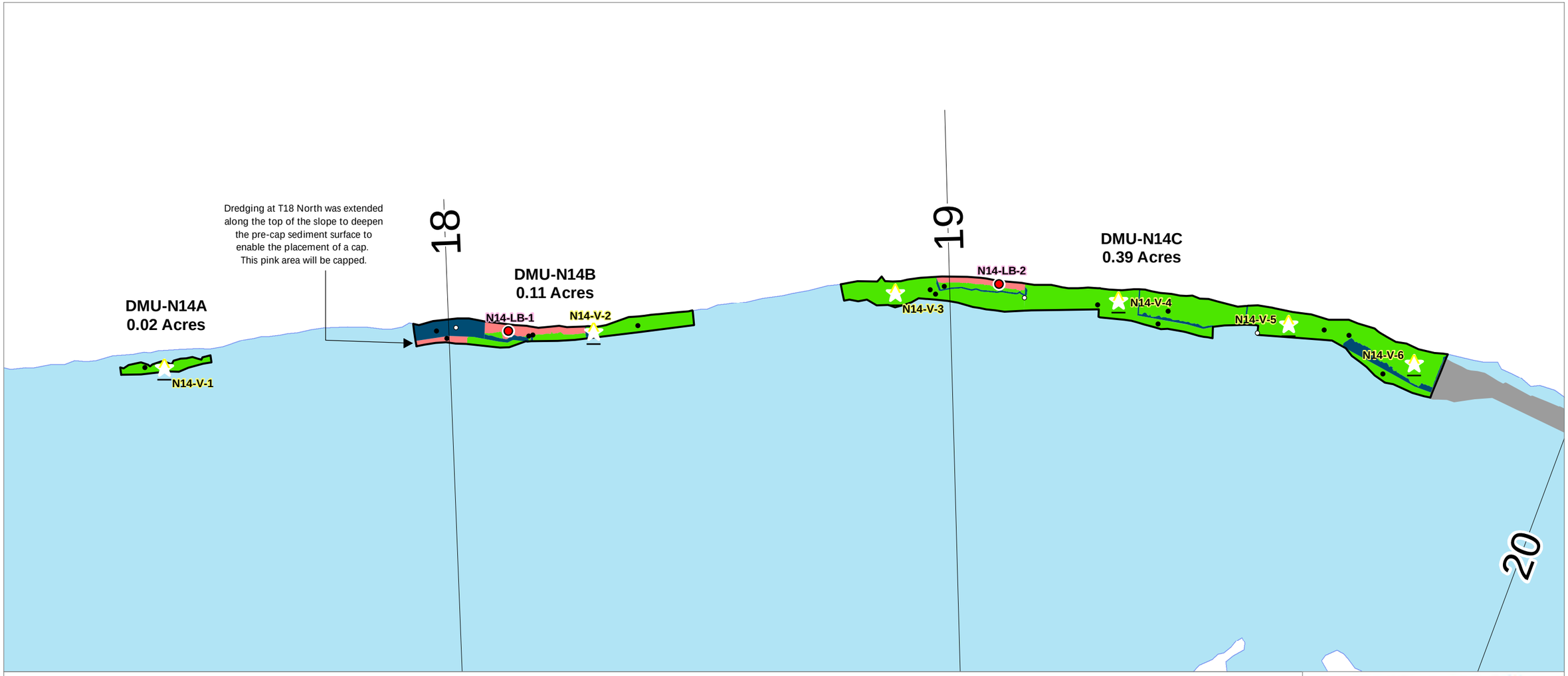

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N14 are shown.
DMU = Dredge Management Unit

0 100
Feet

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ARCONIC

Figure CB-14
Target Post-Dredge Sampling Locations: DMU-N14
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S15 are shown.
DMU = Dredge Management Unit

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Feet

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Figure CB-15
Target Post-Dredge Sampling Locations: DMU-S15
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N16 are shown.
DMU = Dredge Management Unit

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Feet

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Figure CB-16
Target Post-Dredge Sampling Locations: DMU-N16
Construction Quality Assurance Plan
Grasse River Project/Arconic

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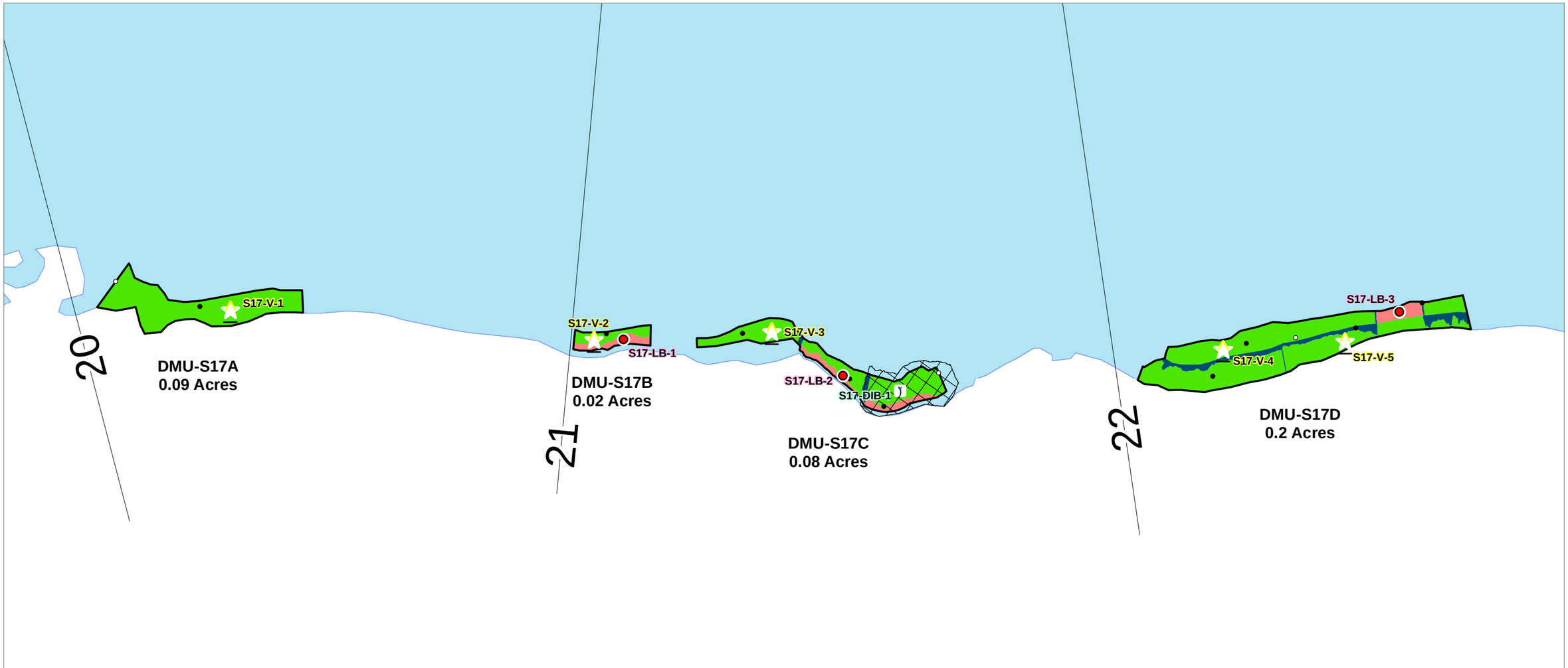

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S17 are shown.
DMU = Dredge Management Unit

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Feet

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ARCONIC

Figure CB-17
Target Post-Dredge Sampling Locations: DMU-S17
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N18 are shown.
DMU = Dredge Management Unit

0 80
Feet

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Figure CB-18
Target Post-Dredge Sampling Locations: DMU-N18
Construction Quality Assurance Plan
Grasse River Project/Arconic

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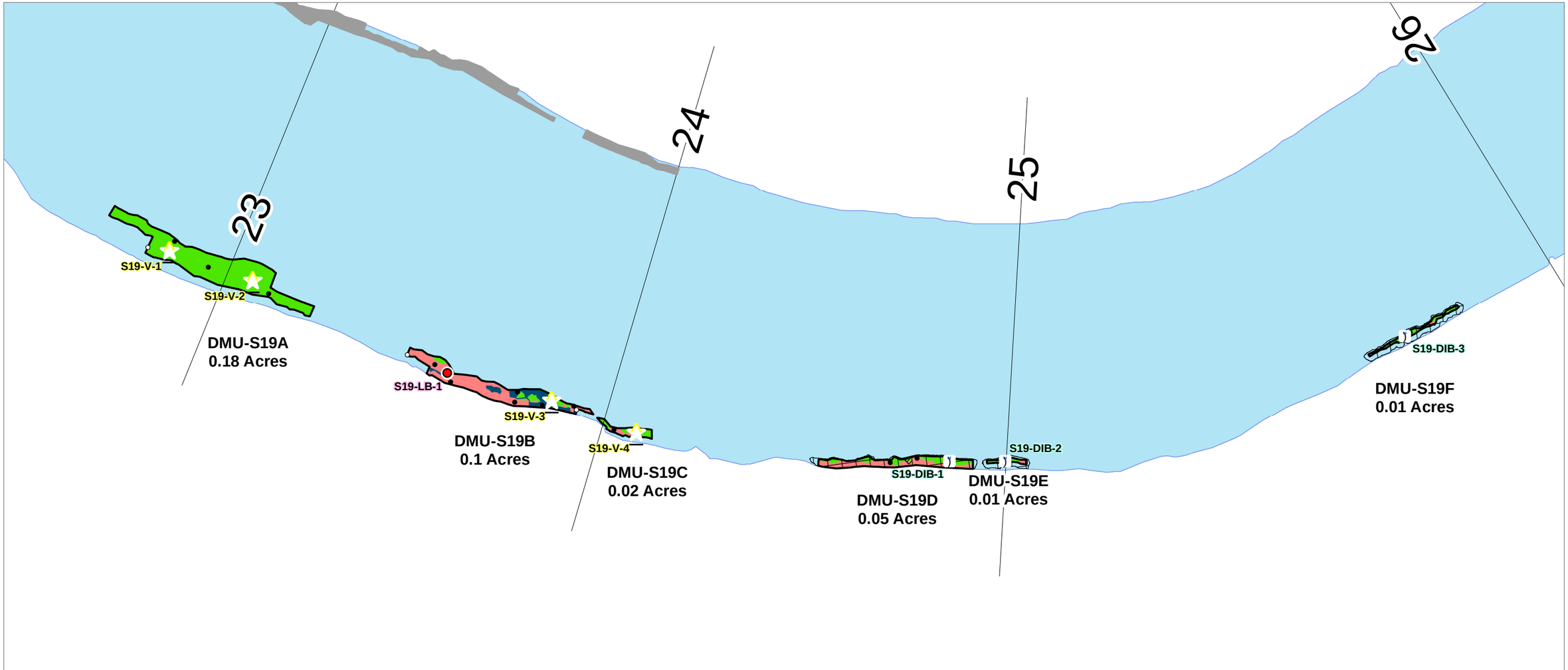

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S19 are shown.
DMU = Dredge Management Unit

0 130
Feet

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ARCONIC

Figure CB-19
Target Post-Dredge Sampling Locations: DMU-S19
Construction Quality Assurance Plan
Grasse River Project/Arconic

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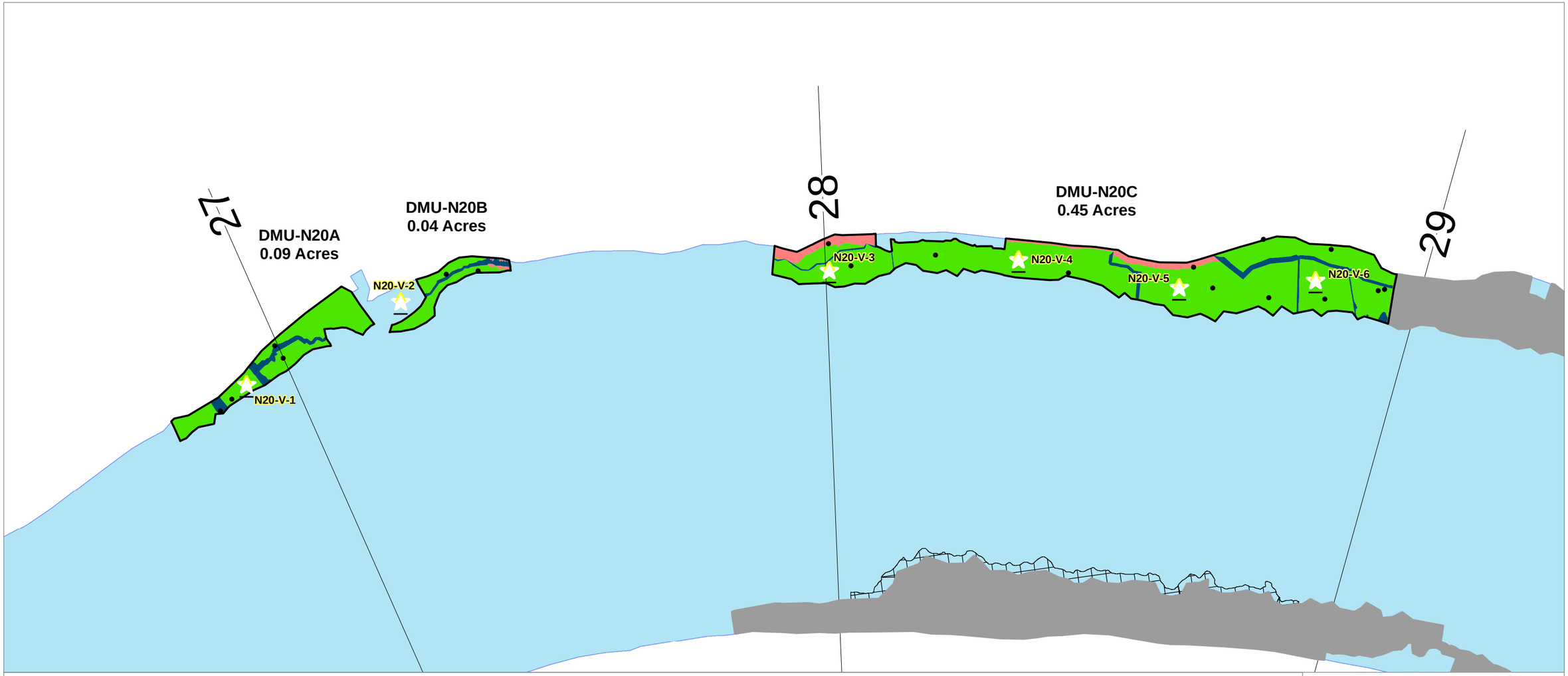

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N20 are shown.
DMU = Dredge Management Unit

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Feet

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ARCONIC

Figure CB-20
Target Post-Dredge Sampling Locations: DMU-N20

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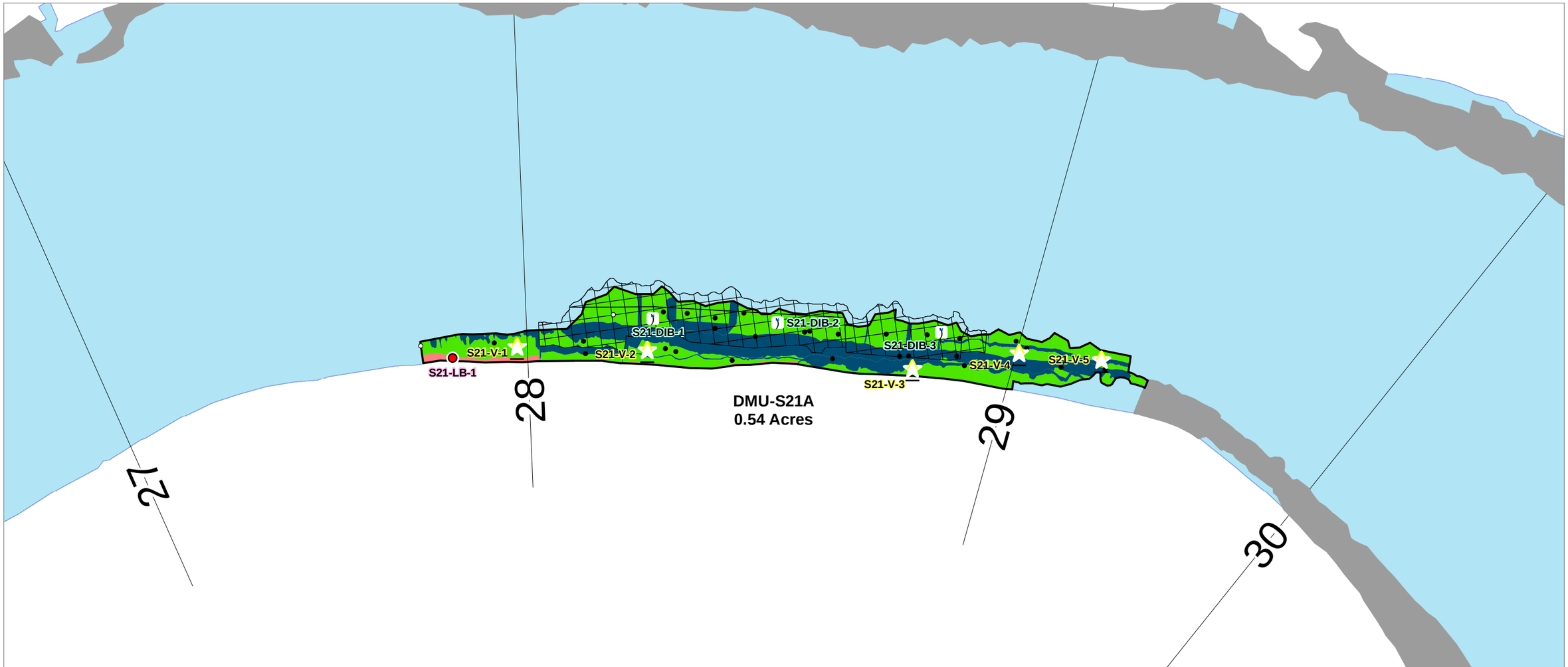

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S21 are shown.
DMU = Dredge Management Unit

0 80
Feet

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Figure CB-21
Target Post-Dredge Sampling Locations: DMU-S21

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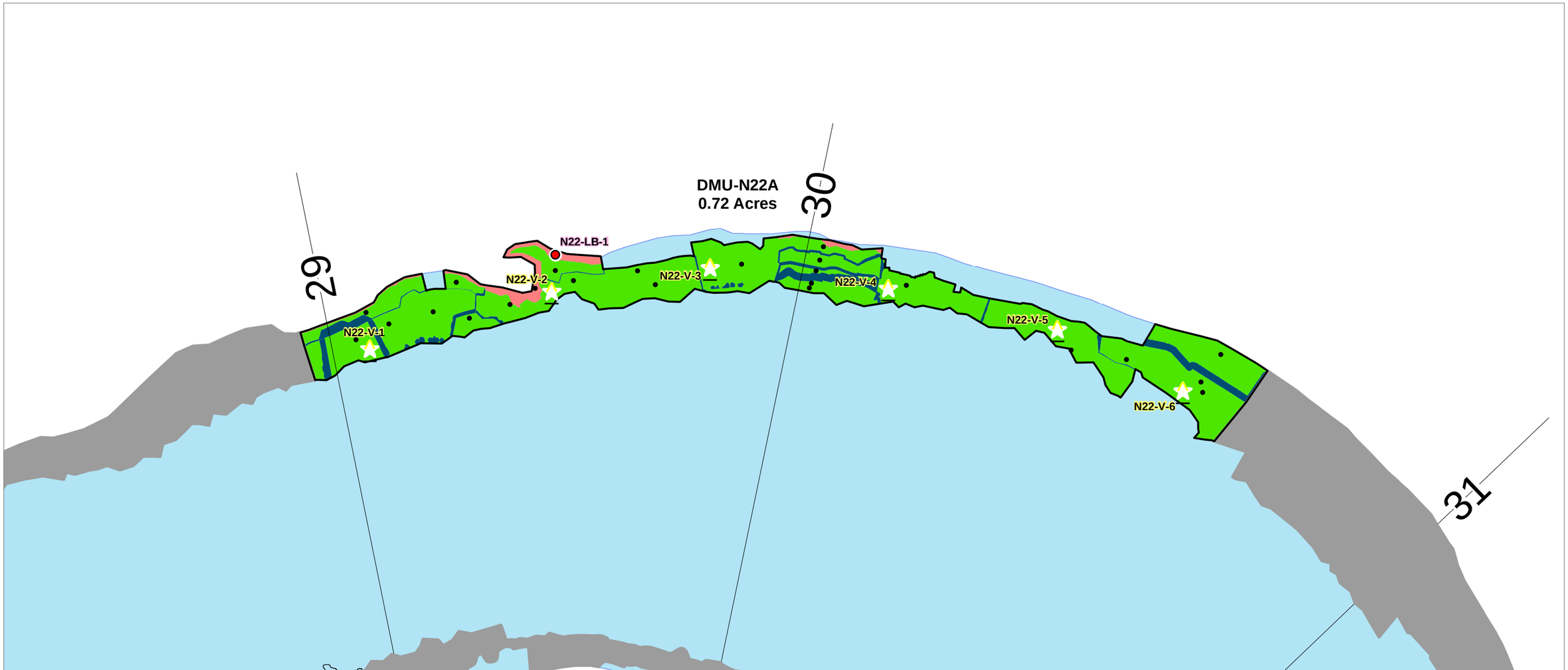

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N22 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-22
Target Post-Dredge Sampling Locations: DMU-N22

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S23 are shown.
DMU = Dredge Management Unit

0 80
Feet

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Figure CB-23
Target Post-Dredge Sampling Locations: DMU-S23
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N24 are shown.
DMU = Dredge Management Unit

0 80
Feet

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Figure CB-24
Target Post-Dredge Sampling Locations: DMU-N24
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S25 are shown.
DMU = Dredge Management Unit

0 80
Feet

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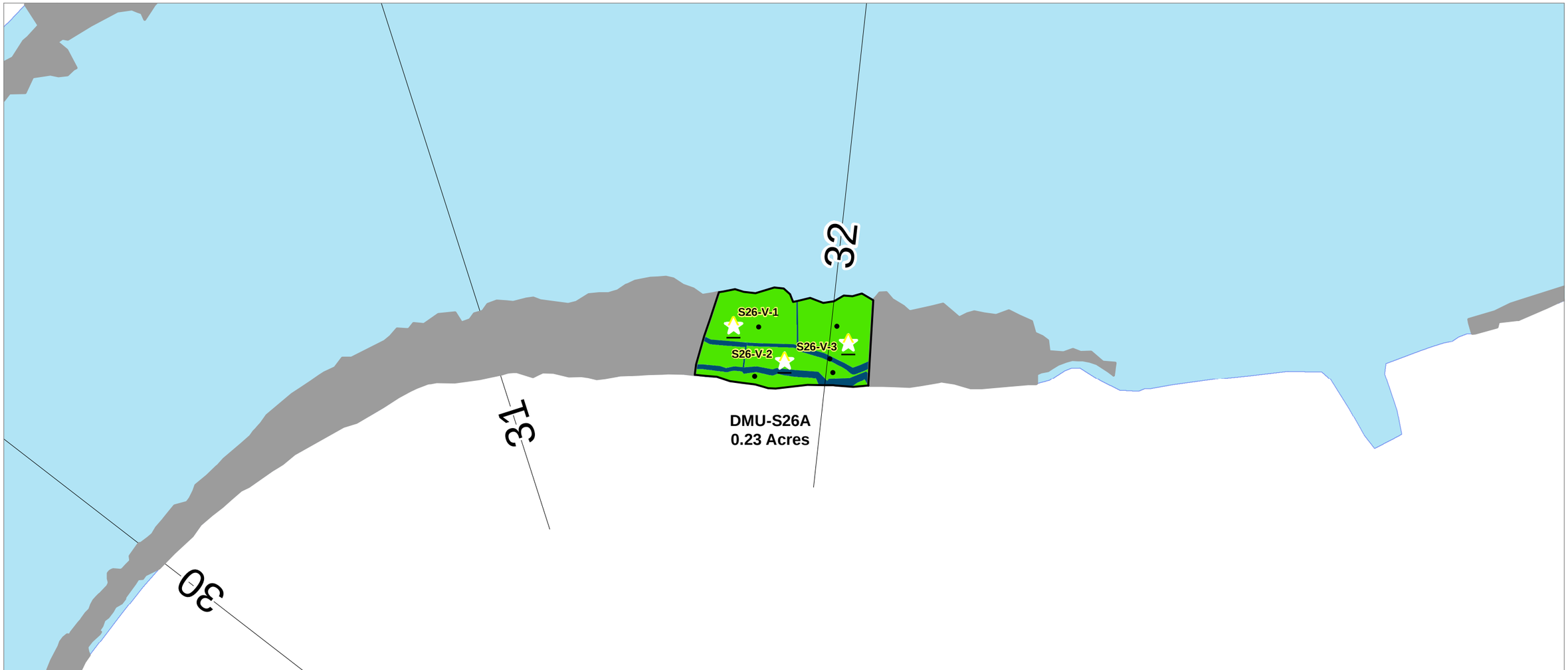
ARCONIC

Figure CB-25
Target Post-Dredge Sampling Locations: DMU-S25
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT



LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S26 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-26
Target Post-Dredge Sampling Locations: DMU-S26
Construction Quality Assurance Plan
Grasse River Project/Arconic

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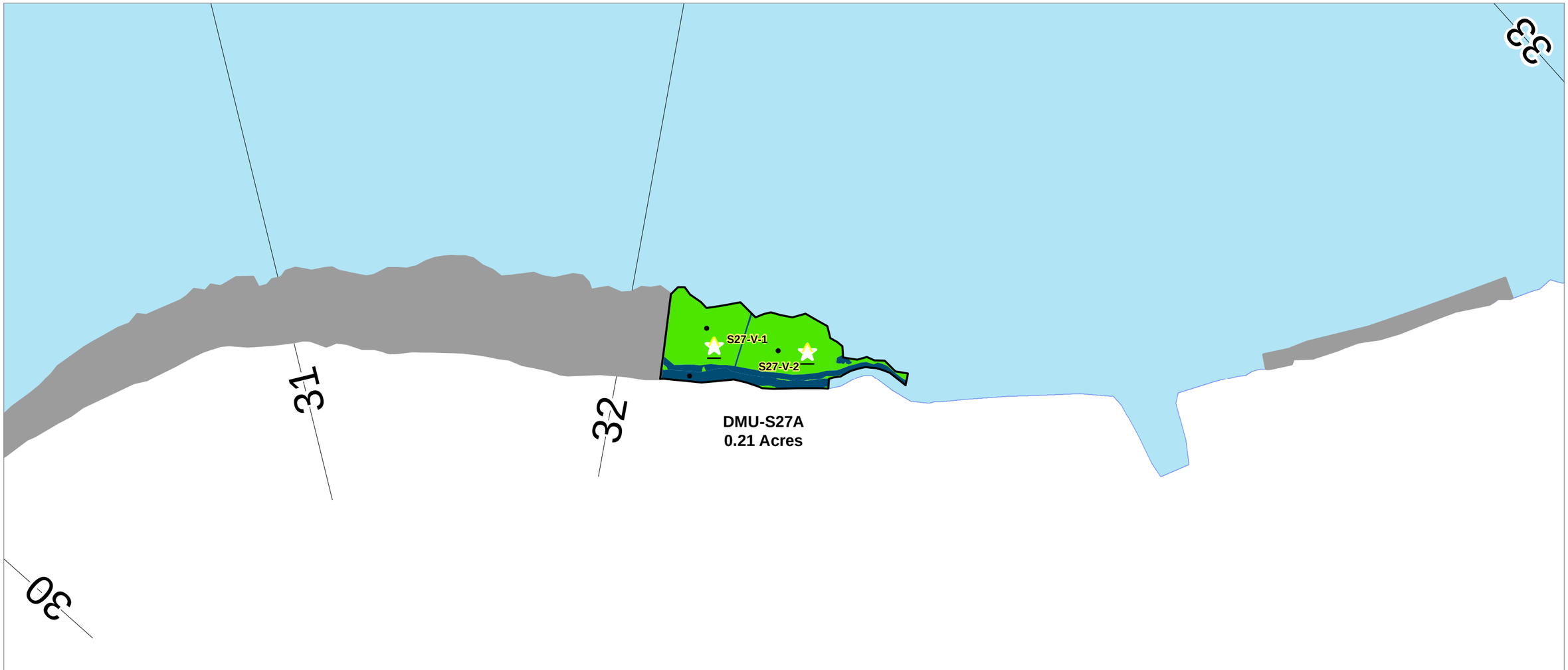

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S27 are shown.
DMU = Dredge Management Unit

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Feet

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ARCONIC

Figure CB-27
Target Post-Dredge Sampling Locations: DMU-S27
Construction Quality Assurance Plan
Grasse River Project/Arconic

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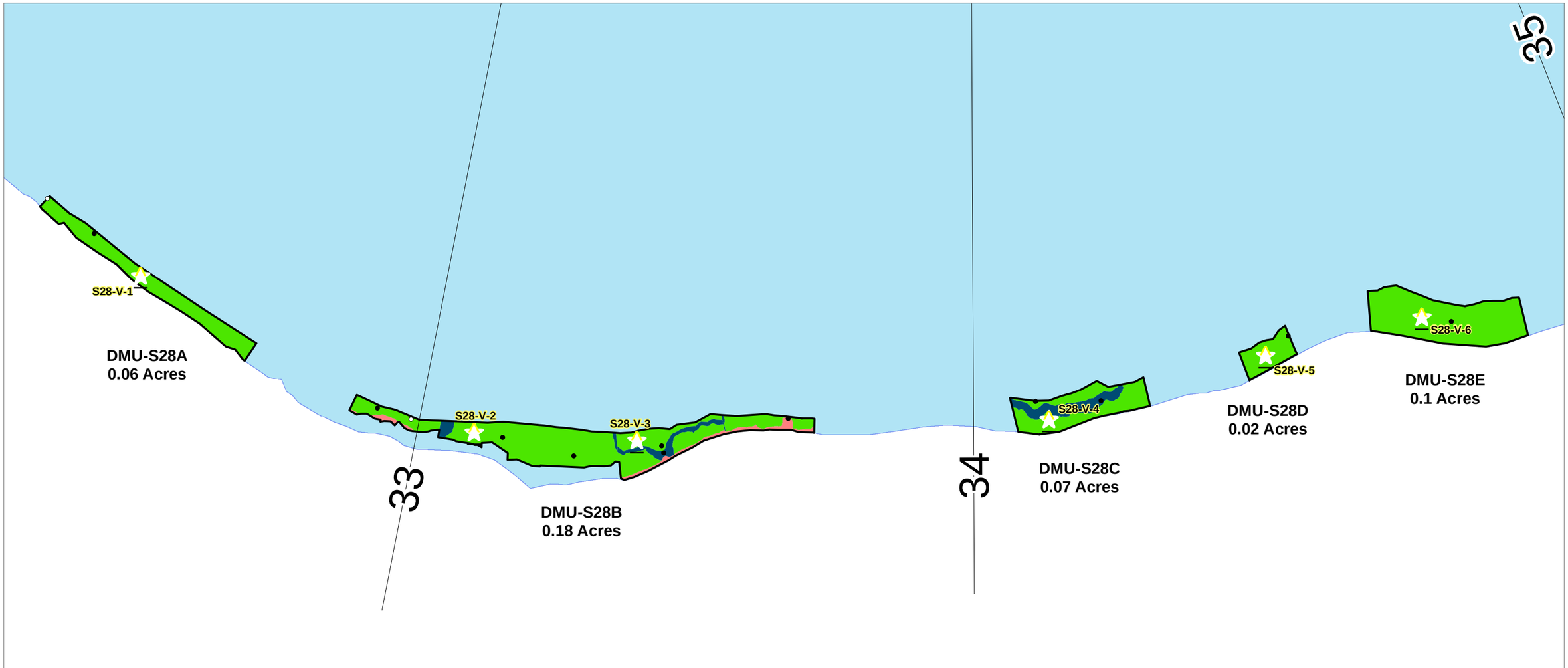

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S28 are shown.
DMU = Dredge Management Unit

0 80
Feet

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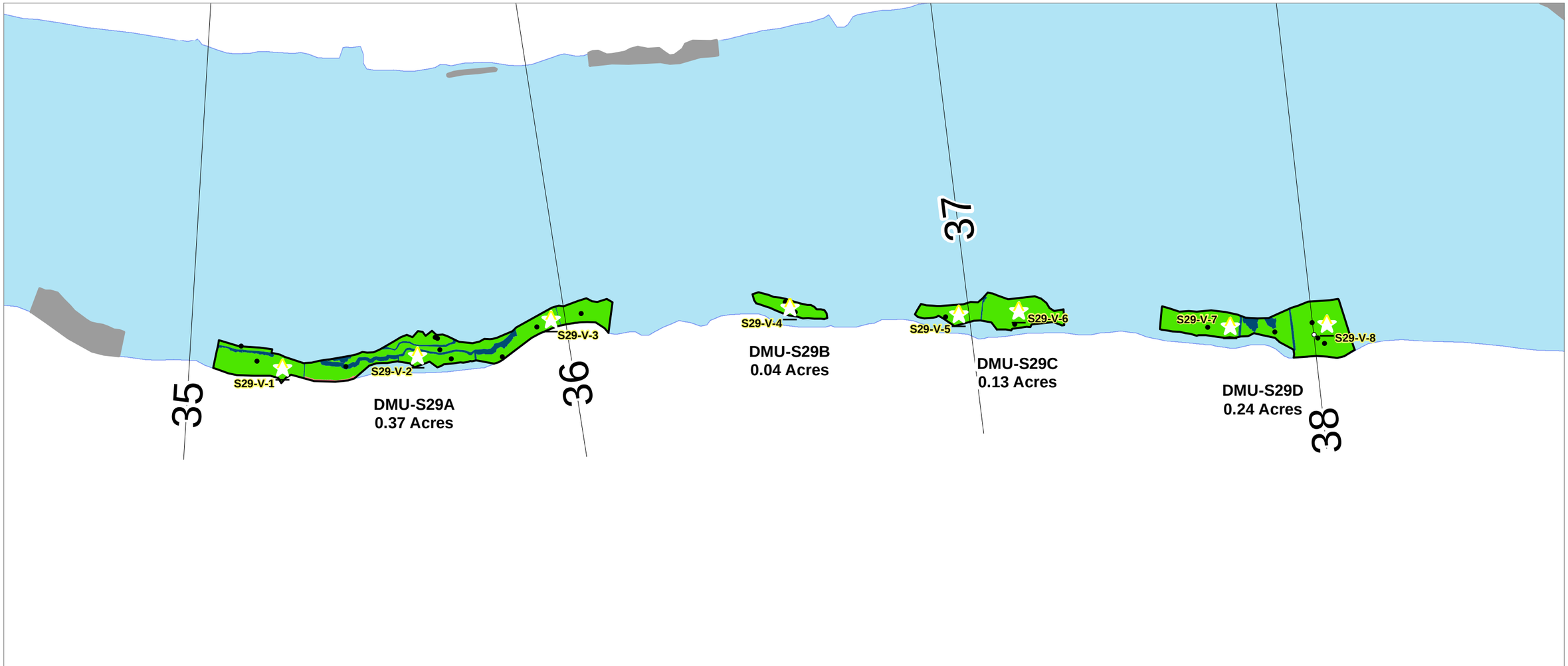
ARCONIC

Figure CB-28
Target Post-Dredge Sampling Locations: DMU-S28
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT



LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S29 are shown.
DMU = Dredge Management Unit

0 130
Feet

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ARCONIC

Figure CB-29
Target Post-Dredge Sampling Locations: DMU-S29
Construction Quality Assurance Plan
Grasse River Project/Arconic

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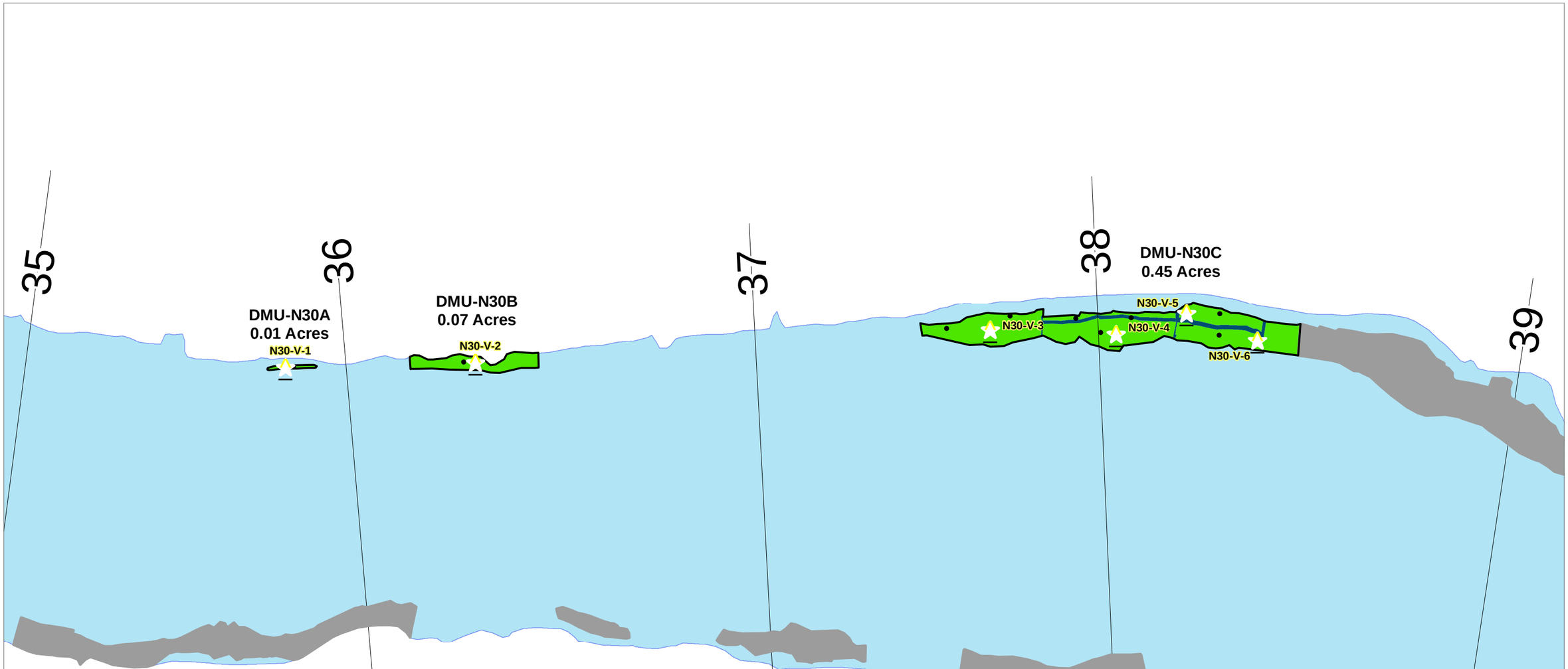

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N30 are shown.
DMU = Dredge Management Unit

0 130
Feet

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ARCONIC

Figure CB-30
Target Post-Dredge Sampling Locations: DMU-N30

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N31 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-31
Target Post-Dredge Sampling Locations: DMU-N31
Construction Quality Assurance Plan
Grasse River Project/Arconic

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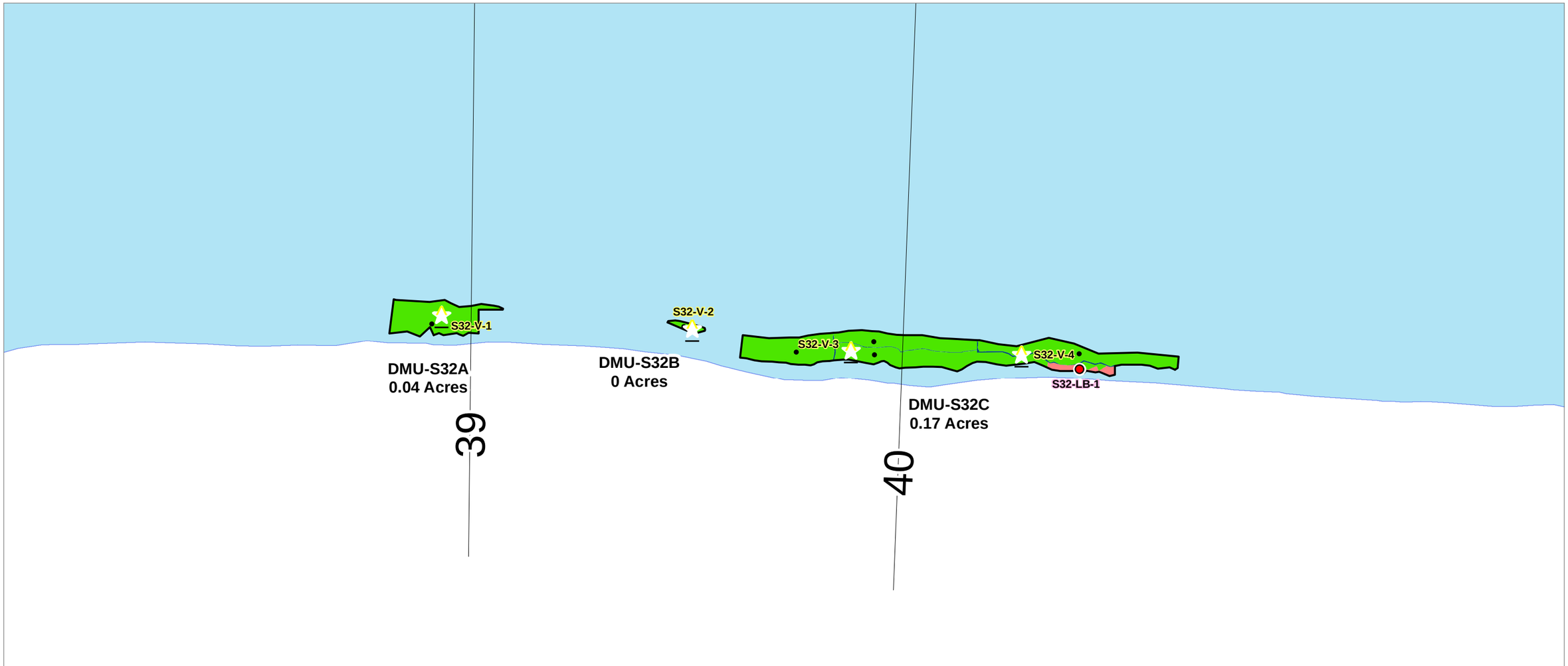

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S32 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-32
Target Post-Dredge Sampling Locations: DMU-S32
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N33 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-33
Target Post-Dredge Sampling Locations: DMU-N33
Construction Quality Assurance Plan
Grasse River Project/Arconic

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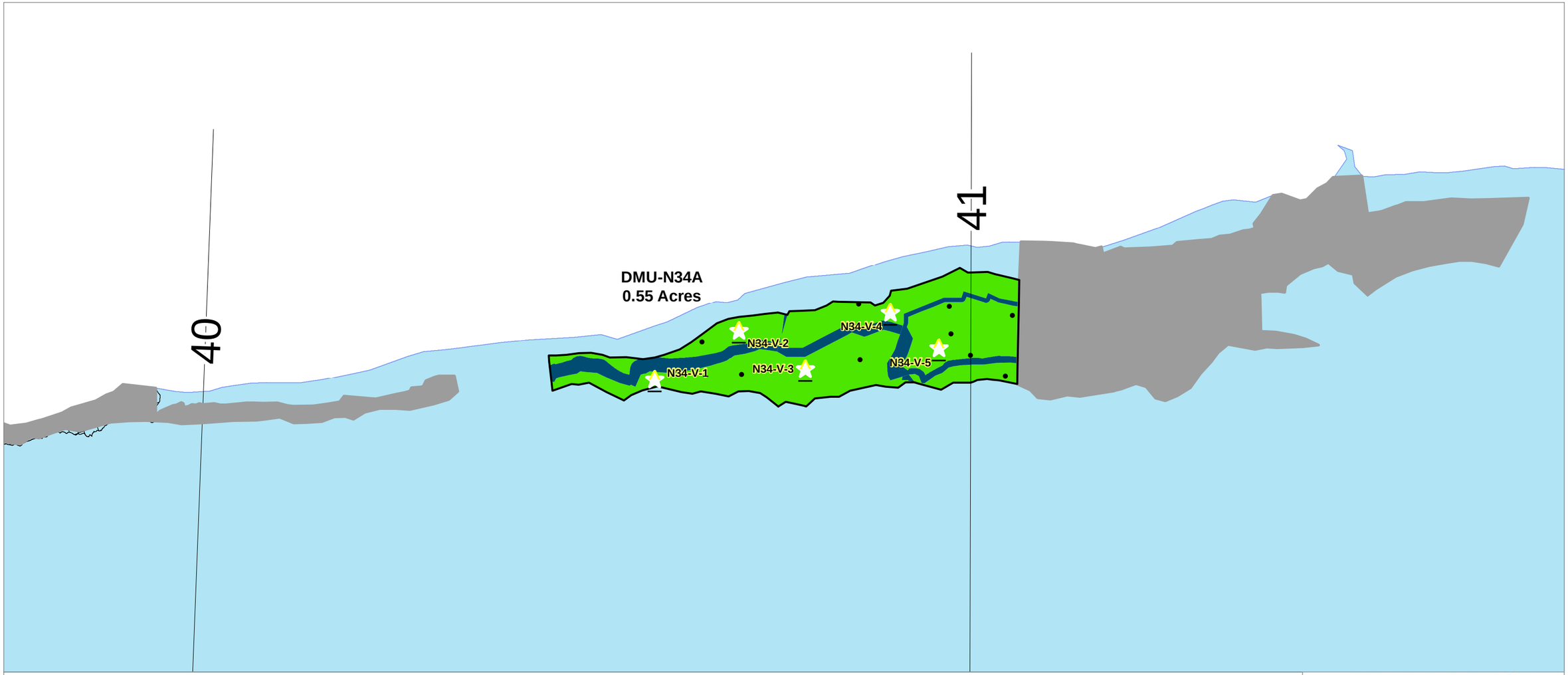

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N34 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-34
Target Post-Dredge Sampling Locations: DMU-N34
Construction Quality Assurance Plan
Grasse River Project/Arconic

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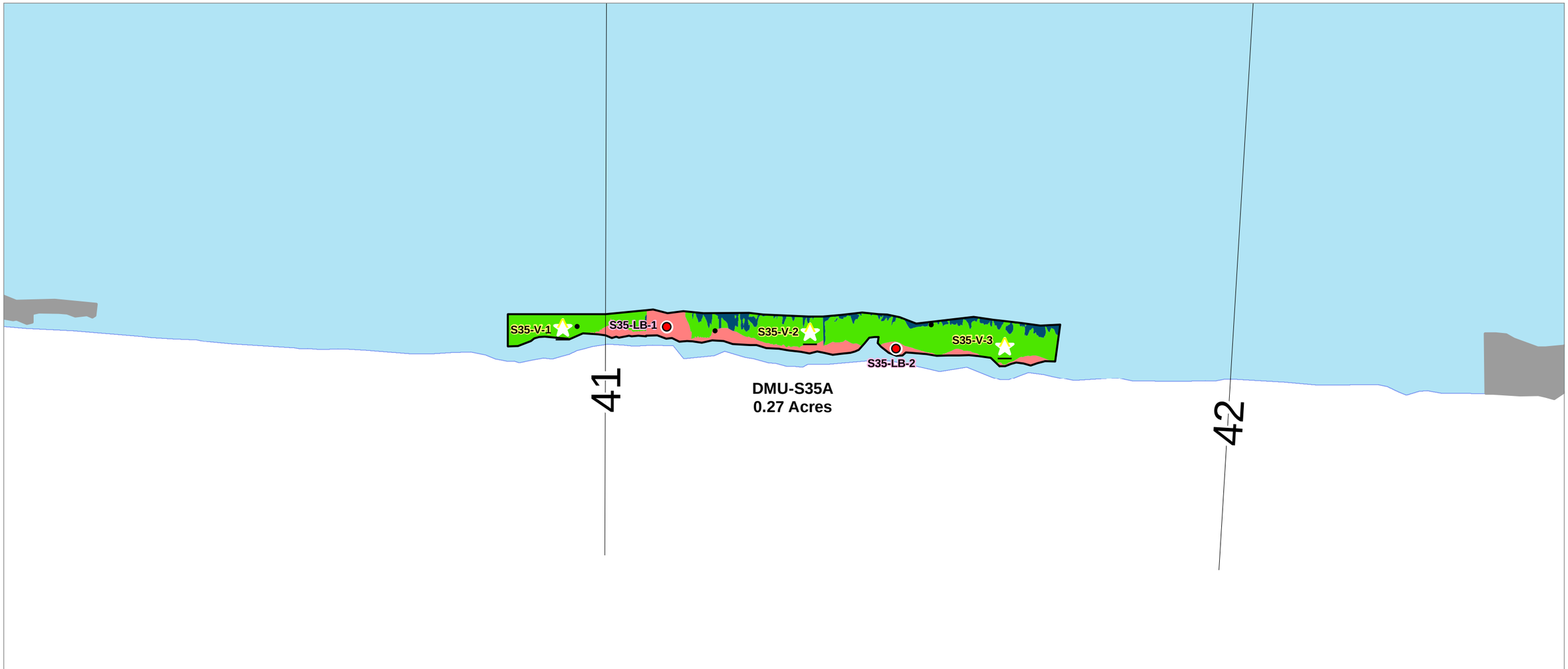

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S35 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-35
Target Post-Dredge Sampling Locations: DMU-S35
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N36 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-36
Target Post-Dredge Sampling Locations: DMU-N36

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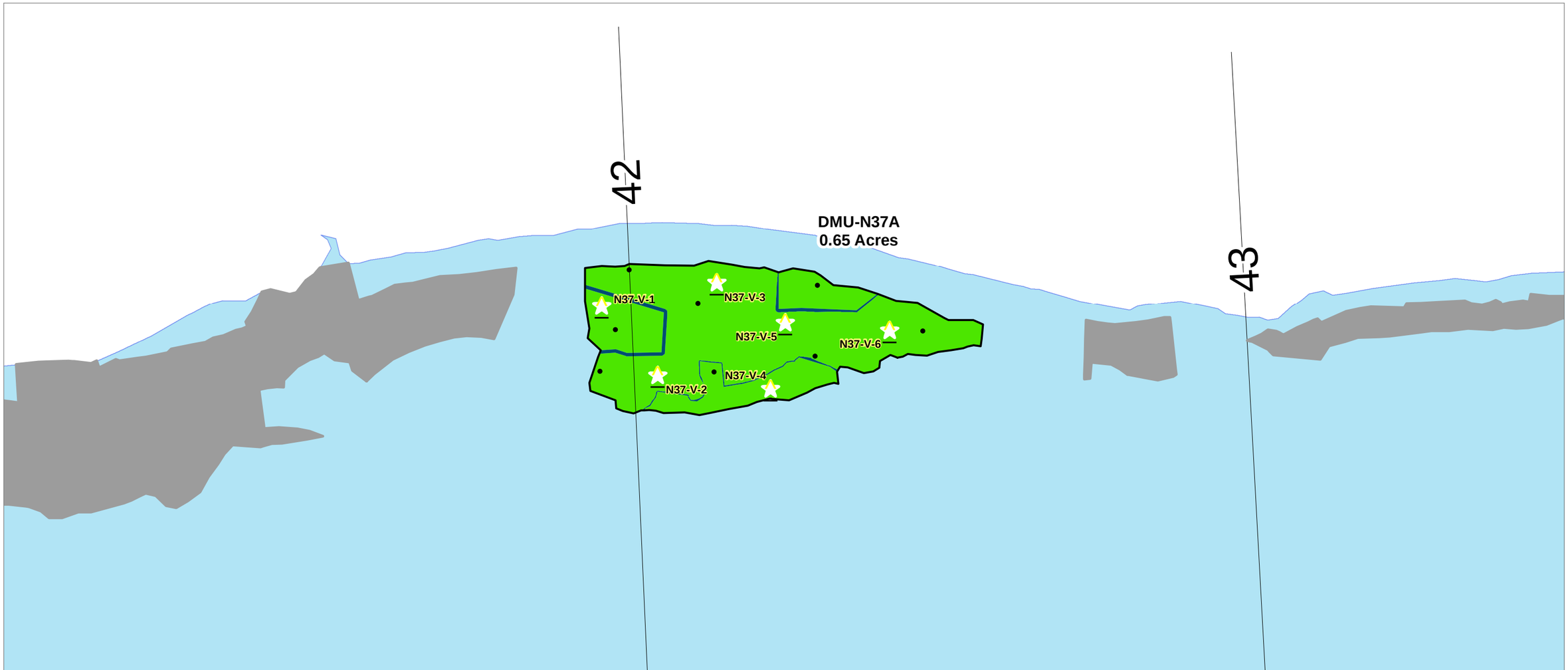

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N37 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-37
Target Post-Dredge Sampling Locations: DMU-N37

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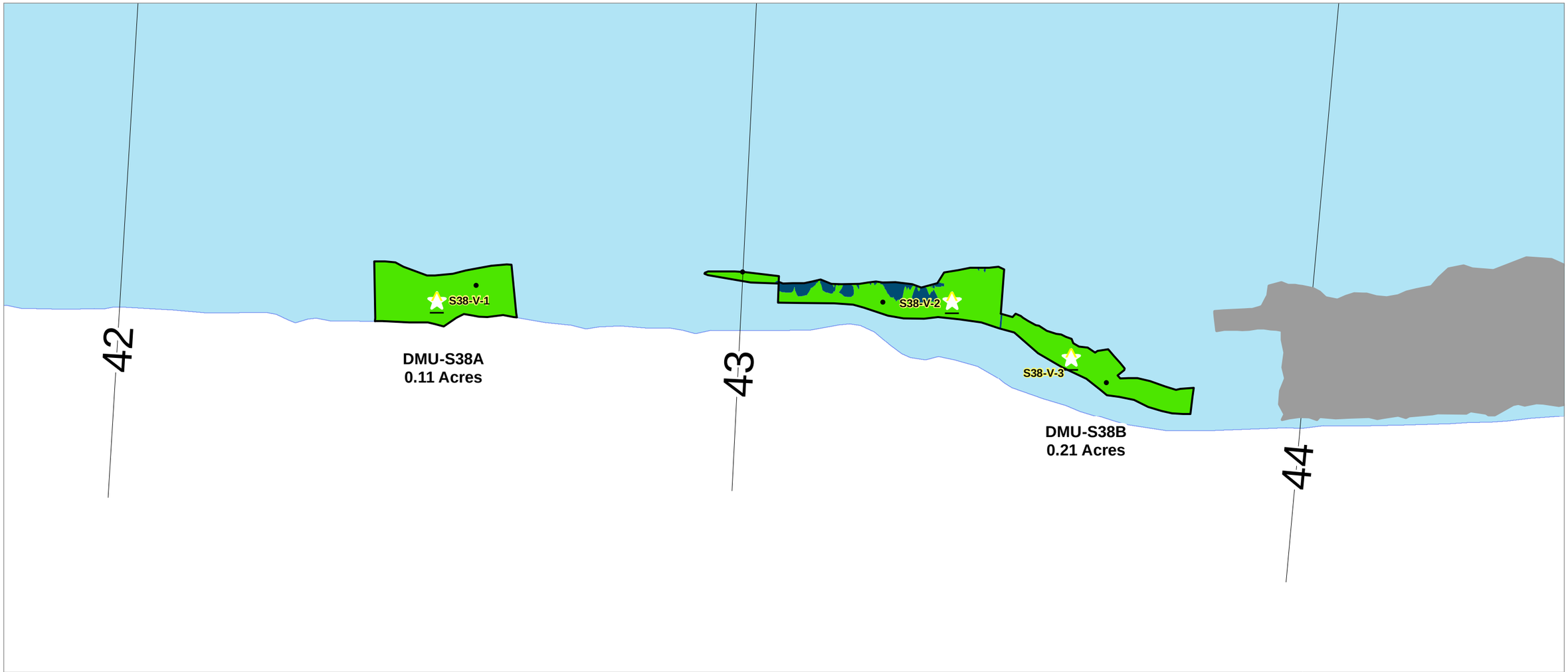

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S38 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-38
Target Post-Dredge Sampling Locations: DMU-S38
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N39 are shown.
DMU = Dredge Management Unit

0 80
Feet

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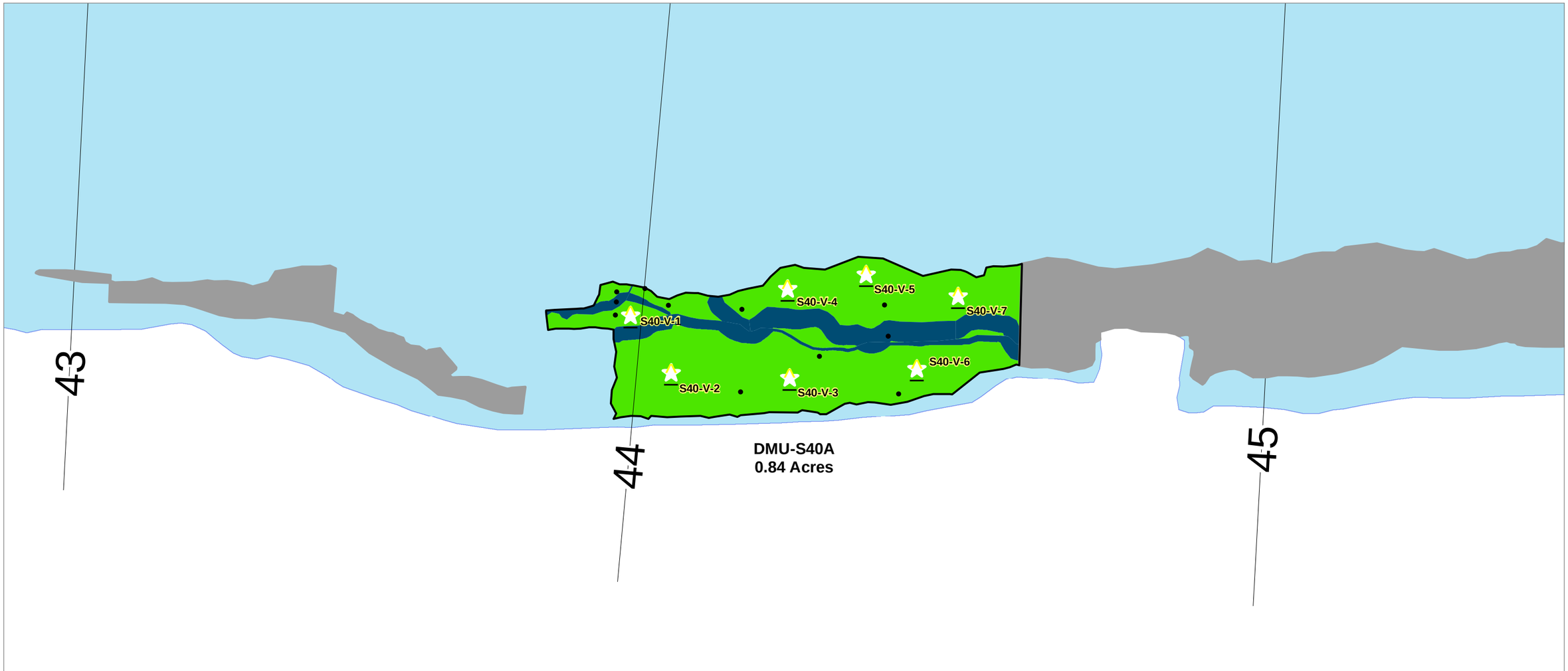
ARCONIC

Figure CB-39
Target Post-Dredge Sampling Locations: DMU-N39
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT



LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S40 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-40
Target Post-Dredge Sampling Locations: DMU-S40
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

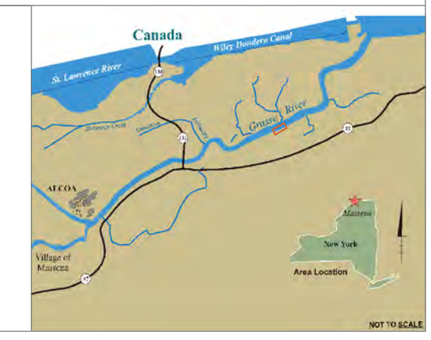
- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S41 are shown.
DMU = Dredge Management Unit

0 80
Feet



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ARCONIC

Figure CB-41
Target Post-Dredge Sampling Locations: DMU-S41
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S42 are shown.
DMU = Dredge Management Unit

0 80
Feet

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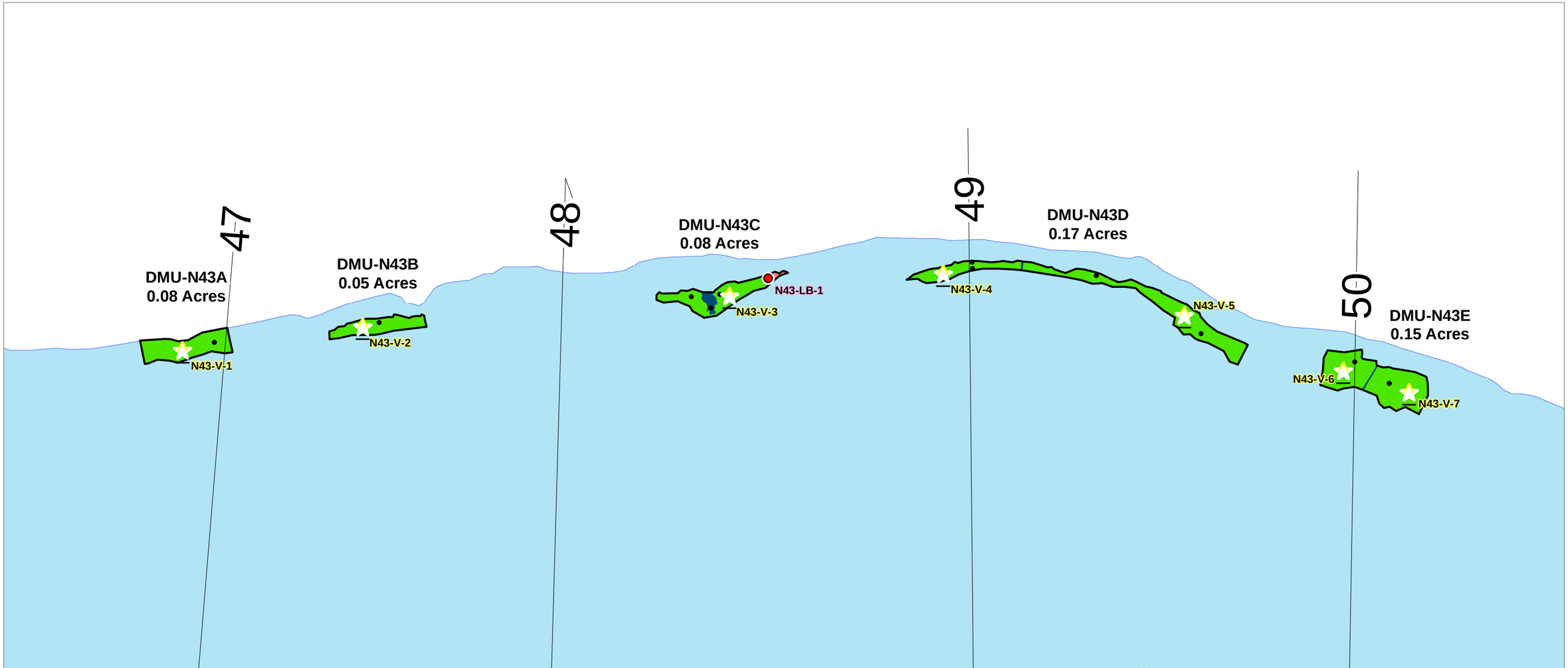
ARCONIC

Figure CB-42
Target Post-Dredge Sampling Locations: DMU-S42

DRAFT



LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N43 are shown.
DMU = Dredge Management Unit

0 130
Feet

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ARCONIC

Figure CB-43
Target Post-Dredge Sampling Locations: DMU-N43
Construction Quality Assurance Plan
Grasse River Project/Arconic

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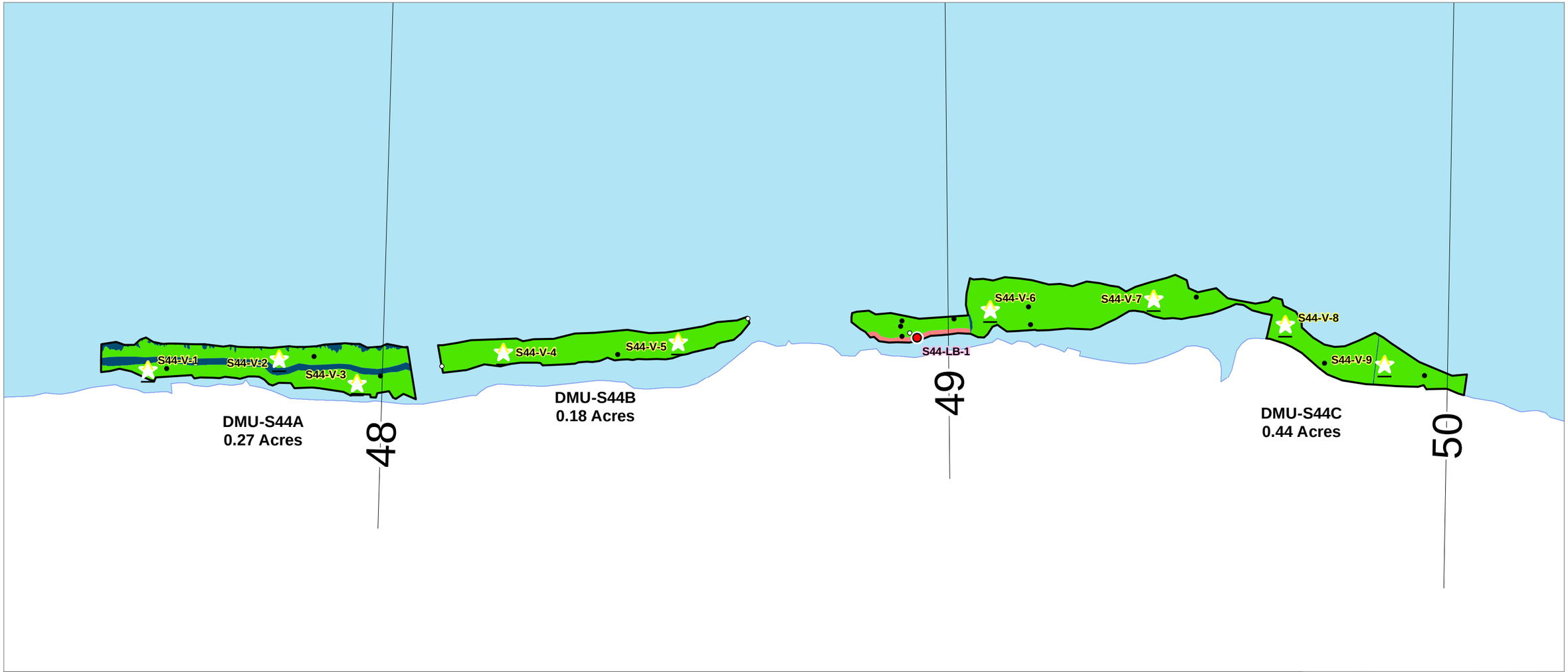

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S44 are shown.
DMU = Dredge Management Unit

0 100
Feet

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ARCONIC

Figure CB-44
Target Post-Dredge Sampling Locations: DMU-S44

DRAFT

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N45 are shown.
DMU = Dredge Management Unit

0 80
Feet

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Figure CB-45
Target Post-Dredge Sampling Locations: DMU-N45
Construction Quality Assurance Plan
Grasse River Project/Arconic

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LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S46 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-46
Target Post-Dredge Sampling Locations: DMU-S46

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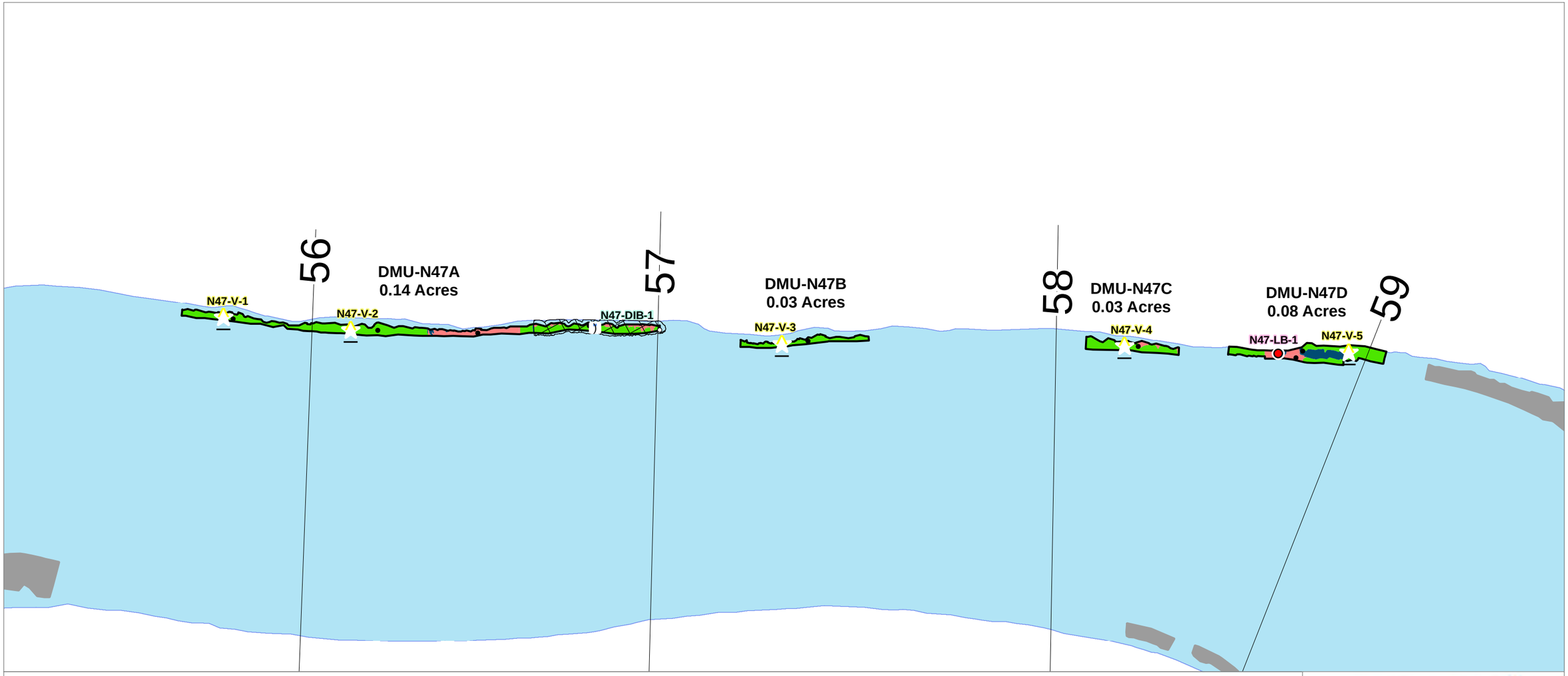

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N47 are shown.
DMU = Dredge Management Unit

0 130
Feet

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ARCONIC

Figure CB-47
Target Post-Dredge Sampling Locations: DMU-N47
Construction Quality Assurance Plan
Grasse River Project/Arconic

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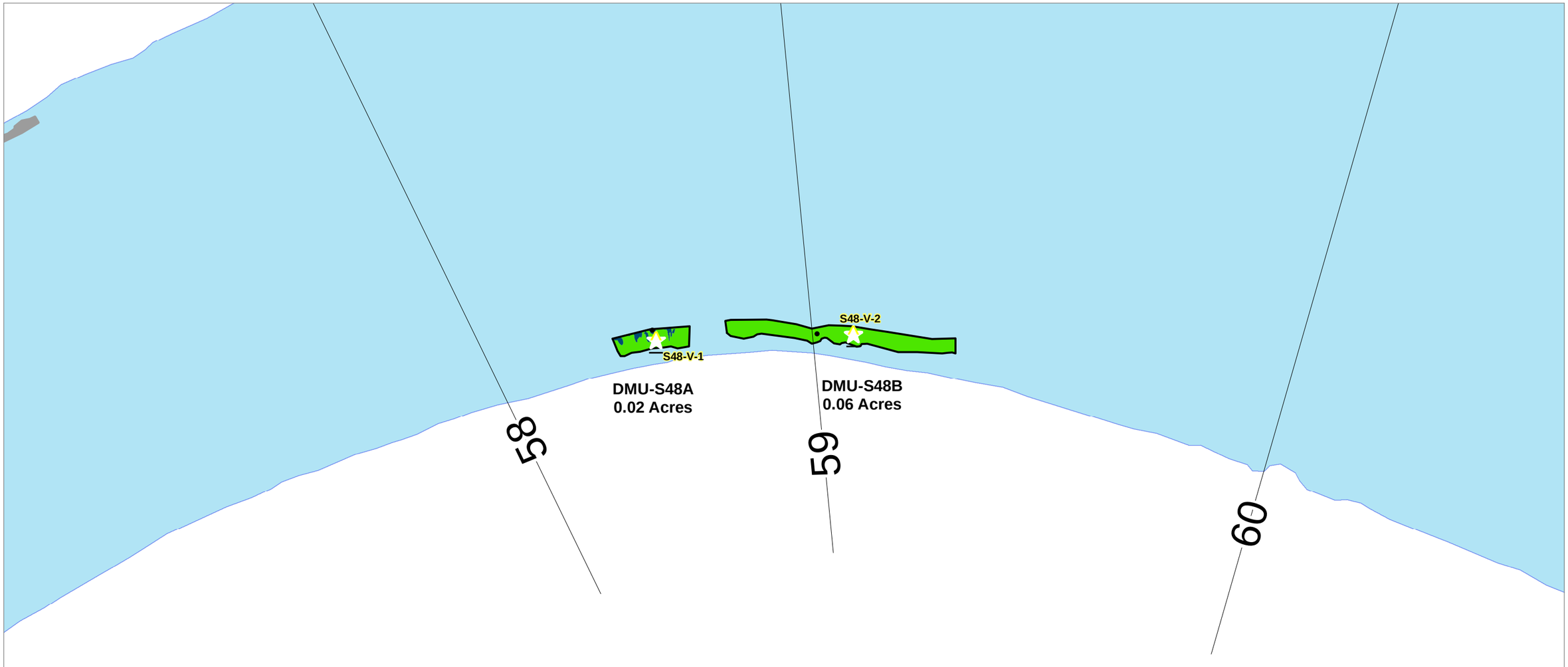

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S48 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-48
Target Post-Dredge Sampling Locations: DMU-S48
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N49 are shown.
DMU = Dredge Management Unit

0 80
Feet

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ARCONIC

Figure CB-49
Target Post-Dredge Sampling Locations: DMU-N49

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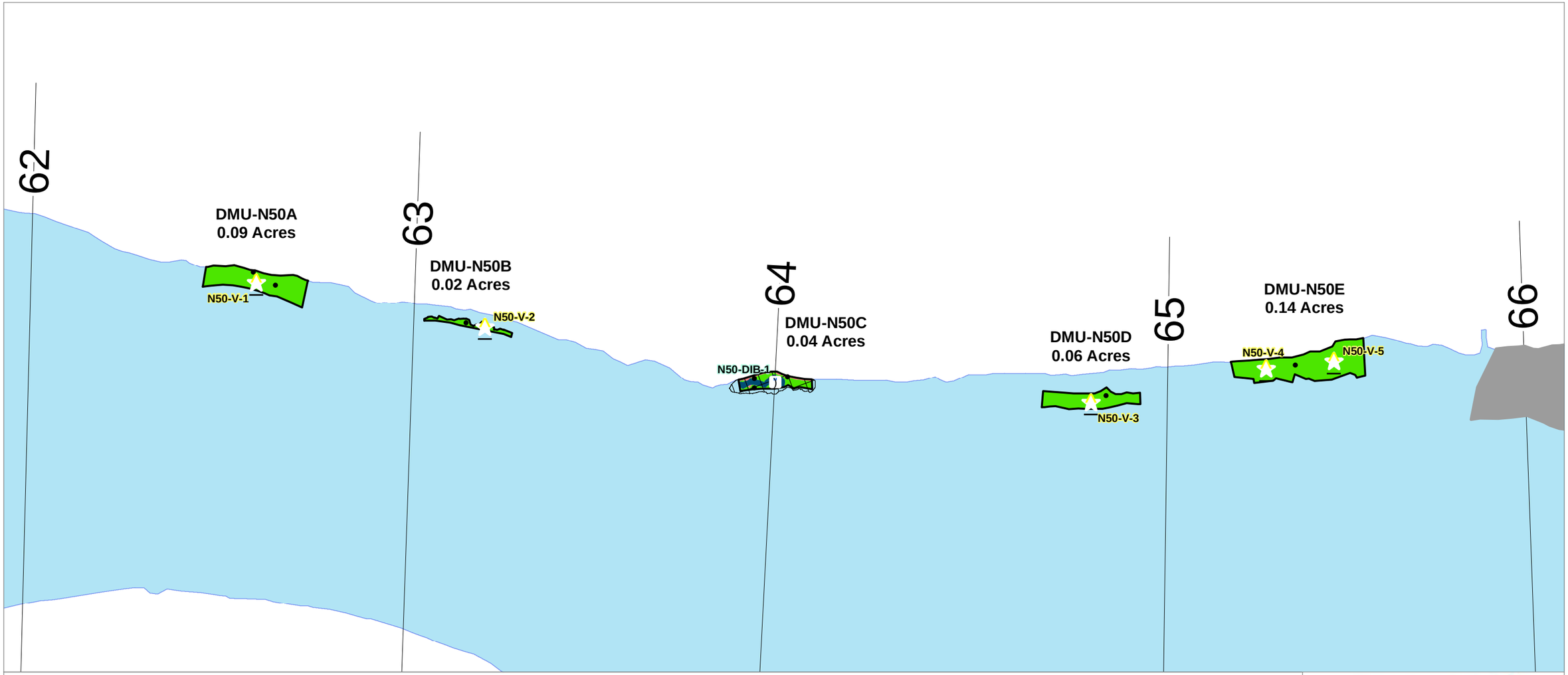


LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

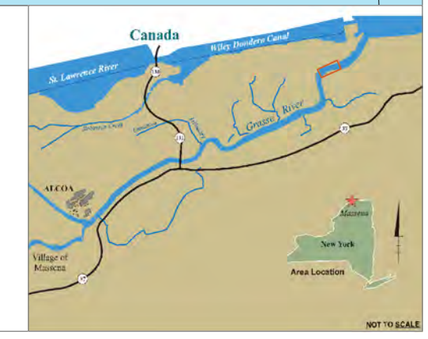
- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N50 are shown.
DMU = Dredge Management Unit

0 130
Feet



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ARCONIC

Figure CB-50
Target Post-Dredge Sampling Locations: DMU-N50
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S51 are shown.
DMU = Dredge Management Unit

0 80
Feet

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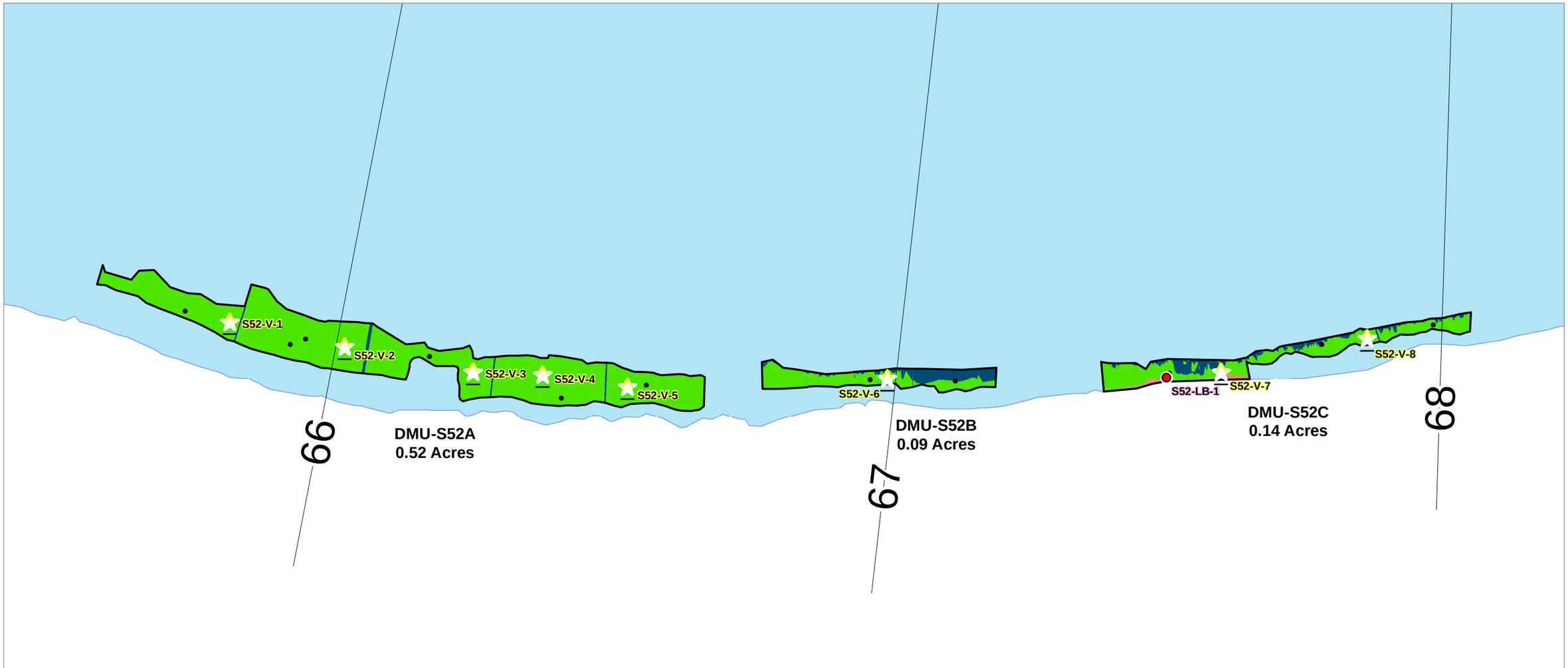
ARCONIC

Figure CB-51
Target Post-Dredge Sampling Locations: DMU-S51

DRAFT



LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S52 are shown.
DMU = Dredge Management Unit

0 100
Feet

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ARCONIC

Figure CB-52
Target Post-Dredge Sampling Locations: DMU-S52
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N53 are shown.
DMU = Dredge Management Unit

0 80
Feet

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Figure CB-53
Target Post-Dredge Sampling Locations: DMU-N53

DRAFT

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N54 are shown.
DMU = Dredge Management Unit

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Feet

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ARCONIC

Figure CB-54
Target Post-Dredge Sampling Locations: DMU-N54

DRAFT

LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-N55 are shown.
DMU = Dredge Management Unit

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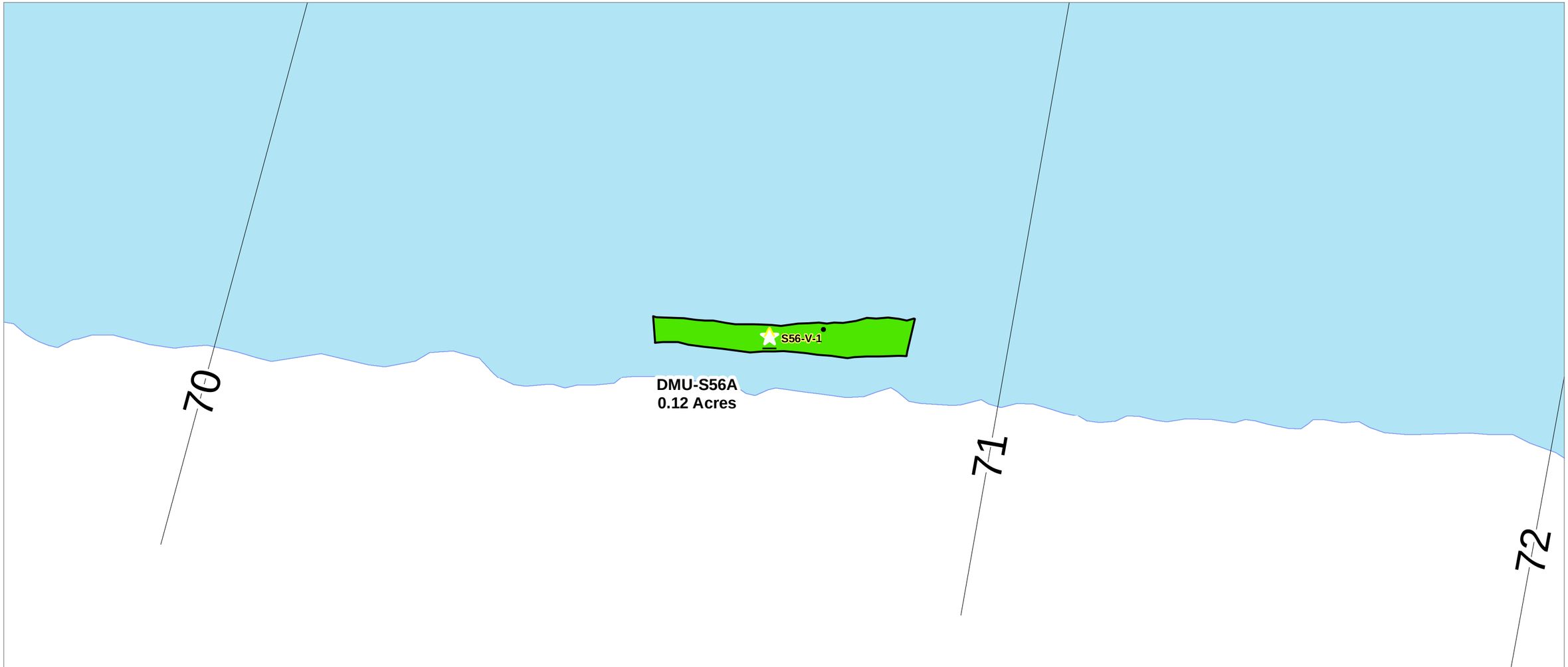
ARCONIC

Figure CB-55
Target Post-Dredge Sampling Locations: DMU-N55
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT



LEGEND:

- DMU Boundary
- Adjacent DMUs
- Dredge & Immediate Backfill Extent
- Grasse River
- Grasse River Transects

Dredge Grading Type (see Note 1)

- Prism Exceeds Chemistry-based Target Dredge Depth
- Prism Meets Chemistry-based Target Dredge Depth
- Dredge Depths Limited by Geotechnical Considerations

Dredge Verification Samples

- Dredge Verification
- Material Left Behind (Documentation Only)
- Dredge & Immediate Backfill (Documentation Only)

PDI Sediment Sampling Locations

- Above Criteria
- Below Criteria
- No Recovery

NOTES:
1. Dredge grading types indicate the engineered dredge prisms target elevation relative to the chemistry-based neatline. The grading of the engineered dredge prism included geotechnical considerations for slope stability, which may result in undercutting or additional removal of material beyond the chemistry-based neatline.
2. Dredge extents have been revised since the draft FDR (September 2018) to reflect design refinements in localized areas where caps would otherwise be placed in shallow water.
3. Proposed DMUs prepared by Anchor QEA and Brennan.
4. Post-dredge sampling location IDs are subject to change.
5. PDI Sediment Sampling Locations within 50 feet of DMU-S56 are shown.
DMU = Dredge Management Unit

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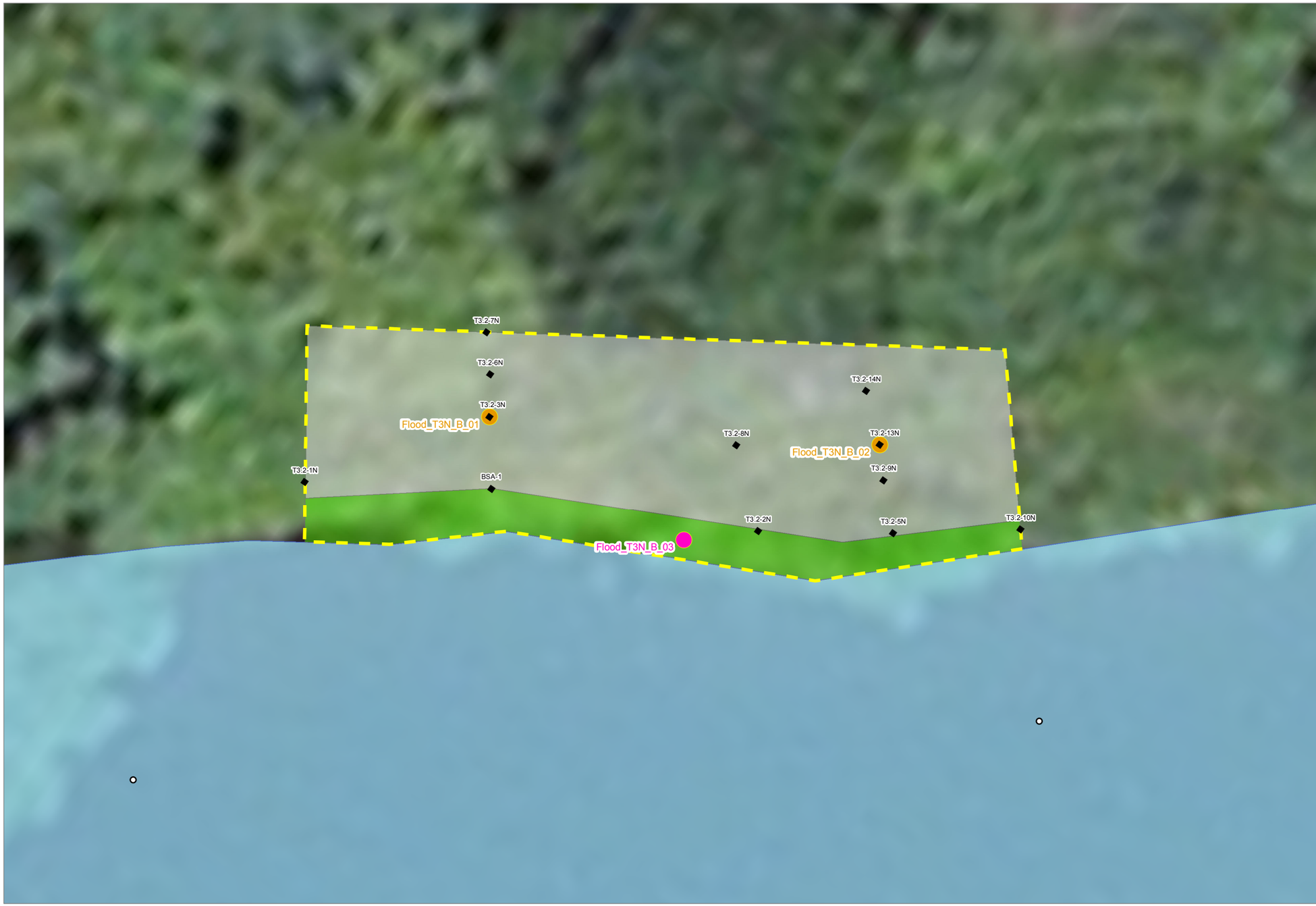
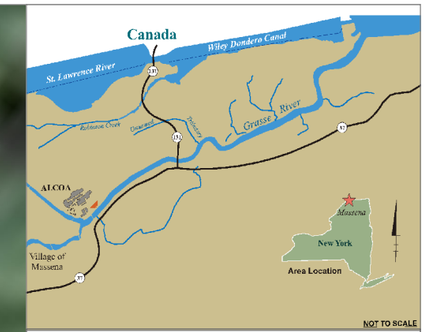
ARCONIC

Figure CB-56
Target Post-Dredge Sampling Locations: DMU-S56
Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT





Floodplain Remedial Area Flood-T3.2N:

Verification Location Count:	**1**
Approx. remedial footprint:	**281 sq. feet**
Target removal depth:	**1.5 feet**
Estimated removal volume:	**16 cubic yards**

LEGEND:

Post-Construction Bottom Sample Locations

- Completed 2018
- Proposed 2019
- Design Sampling Locations
- Floodplain Remedial Extent

Floodplain Excavation Status

- Excavated from Upland in 2018
- Excavate from the River in 2019

Sediment Sampling Locations

- Above Dredge Criteria
- Below Dredge Criteria
- No Recovery
- Final Design Dredge Areas
- Grasse River
- Grasse RiverTransects

NOTES:
Grasse River shoreline extent is based on the high-water elevation of 155.5 feet (USLS 1935).
Floodplain design and proposed verification locations are each labeled with their respective location IDs.
Final Design Dredge Areas are labeled with the dredge DoC (in) or EoC (ft).

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ARCONIC

Figure CB-57
Floodplain Remedial Verification Sample Locations - Flood-T3.2N

DRAFT

Floodplain Remedial Area Flood-T4N:

Verification Location Count:	**2**
Approx. remedial footprint:	**1377 sq. feet**
Target removal depth:	**1.5 feet**
Estimated removal volume:	**77 cubic yards**

LEGEND:

Post-Construction Bottom Sample Locations

- Completed 2018
- Proposed 2019
- Design Sampling Locations
- Floodplain Remedial Extent

Floodplain Excavation Status

- Excavated from Upland in 2018
- Excavate from the River in 2019

Sediment Sampling Locations

- Above Dredge Criteria
- Below Dredge Criteria
- No Recovery
- Final Design Dredge Areas
- Grasse River
- Grasse RiverTransects

NOTES:
Grasse River shoreline extent is based on the high-water elevation of 155.5 feet (USLS 1935).
Floodplain design and proposed verification locations are each labeled with their respective location IDs.
Final Design Dredge Areas are labeled with the dredge DoC (in) or EoC (ft).

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Feet

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ARCONIC

Figure CB-58
Floodplain Remedial Verification Sample Locations - Flood-T4N

DRAFT

Floodplain Remedial Area Flood-T6.3N:

Verification Location Count:	**2**
Approx. remedial footprint:	**943 sq. feet**
Target removal depth:	**1.5 feet**
Estimated removal volume:	**52 cubic yards**

LEGEND:

Post-Construction Bottom Sample Locations

- Completed 2018
- Proposed 2019
- Design Sampling Locations
- Floodplain Remedial Extent

Floodplain Excavation Status

- Excavated from Upland in 2018
- Excavate from the River in 2019

Sediment Sampling Locations

- Above Dredge Criteria
- Below Dredge Criteria
- No Recovery
- Final Design Dredge Areas
- Grasse River
- Grasse RiverTransects

NOTES:
Grasse River shoreline extent is based on the high-water elevation of 155.5 feet (USLS 1935).
Floodplain design and proposed verification locations are each labeled with their respective location IDs.
Final Design Dredge Areas are labeled with the dredge DoC (in) or EoC (ft).

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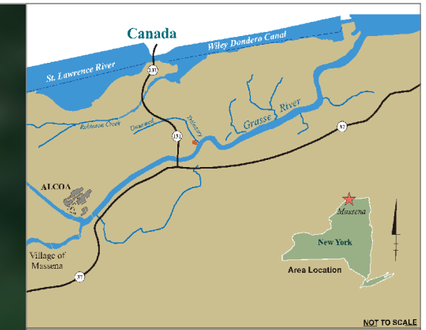
ARCONIC

Figure CB-59
Floodplain Remedial Verification Sample Locations - Flood-T6.3N

Construction Quality Assurance Plan
Grasse River Project/Arconic

DRAFT



Floodplain Remedial Area UNT-T27N:

Verification Location Count:	**15**
Approx. remedial footprint:	**5037 sq. feet**
Target removal depth:	**2 feet**
Estimated removal volume:	**373 cubic yards**

LEGEND:

Post-Construction Bottom Sample Locations

- Completed 2018
- Proposed 2019

Post-Construction Sidewall Sample Locations

- Proposed 2019
- Design Sampling Locations
- Floodplain Remedial Extent

Floodplain Excavation Status

- Excavated from Upland in 2018
- Excavate from the River in 2019

Sediment Sampling Locations

- Above Dredge Criteria
- Below Dredge Criteria
- No Recovery
- Final Design Dredge Areas
- Grasse River
- Grasse RiverTransects

NOTES:
Grasse River shoreline extent is based on the high-water elevation of 155.5 feet (USLS 1935).
Floodplain design and proposed verification locations are each labeled with their respective location IDs.
Final Design Dredge Areas are labeled with the dredge DoC (in) or EoC (ft).

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Feet

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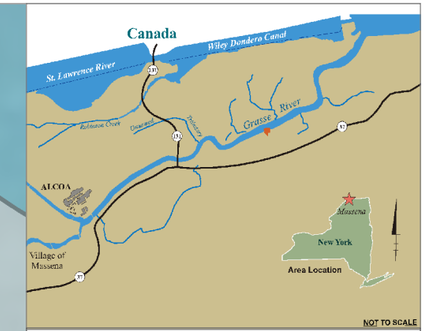
ARCONIC

Figure CB-60
Floodplain Remedial Verification Sample Locations - UNT-T27N

DRAFT



Floodplain Remedial Area Flood-T43.2S:

Verification Location Count:	**15**
Approx. remedial footprint:	**6637 sq. feet**
Target removal depth:	**1.5 feet**
Estimated removal volume:	**369 cubic yards**

LEGEND:

Post-Construction Bottom Sample Locations

- Completed 2018
- Proposed 2019

Post-Construction Sidewall Sample Locations

- Proposed 2019
- Design Sampling Locations
- Floodplain Remedial Extent

Floodplain Excavation Status

- Excavated from Upland in 2018
- Excavate from the River in 2019

Sediment Sampling Locations

- Above Dredge Criteria
- Below Dredge Criteria
- No Recovery
- Final Design Dredge Areas
- Grasse River
- Grasse RiverTransects

NOTES:
Grasse River shoreline extent is based on the high-water elevation of 155.5 feet (USLS 1935).
Floodplain design and proposed verification locations are each labeled with their respective location IDs.
Final Design Dredge Areas are labeled with the dredge DoC (in) or EoC (ft).

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Feet

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ARCONIC

Figure CB-61
Floodplain Remedial Verification Sample Locations - Flood-T43.2S

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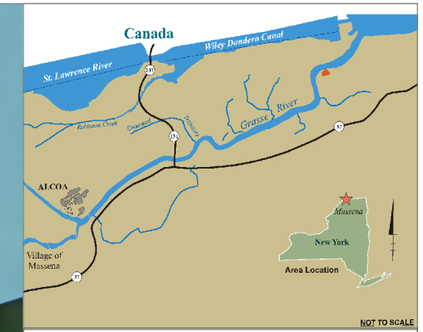

Floodplain Remedial Area Flood-T64S:

Verification Location Count:	**22**
Approx. remedial footprint:	**16392 sq. feet**
Target removal depth:	**1.5 feet**
Estimated removal volume:	**911 cubic yards**

LEGEND:

Post-Construction Bottom Sample Locations

- Completed 2018
- Proposed 2019

Post-Construction Sidewall Sample Locations

- Proposed 2019
- Design Sampling Locations
- Floodplain Remedial Extent

Floodplain Excavation Status

- Excavated from Upland in 2018
- Excavate from the River in 2019

Sediment Sampling Locations

- Above Dredge Criteria
- Below Dredge Criteria
- No Recovery
- Final Design Dredge Areas
- Grasse River
- Grasse RiverTransects

NOTES:
Grasse River shoreline extent is based on the high-water elevation of 155.5 feet (USLS 1935).
Floodplain design and proposed verification locations are each labeled with their respective location IDs.
Final Design Dredge Areas are labeled with the dredge DoC (in) or EoC (ft).

0 40
Feet

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ARCONIC

Figure CB-62
Floodplain Remedial Verification Sample Locations - Flood-T64S

Analytical Standard Operating Procedures

P-07

Determination of Polychlorinated Biphenyls (PCBs) Aroclors (Rev. 03a; 09/27/2016; S-NY-O-314)

STANDARD OPERATING PROCEDURE

DETERMINATION OF POLYCHLORINATED BIPHENYL (PCB) AROCLORS

Reference Methods: EPA Method 8082A

SOP Number:	S-NY-O-314-rev.03a
Effective Date:	09/27/2016
Supersedes:	S-NY-O-314-rev.03

APPROVALS

Joann Slavin General Manager	09-28-16 Date
Roy Smith Quality Manager	09-27-2016 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose/Identification of Method

1.1. This Standard Operating Procedure (SOP) is used to determine Polychlorinated Biphenyl (PCB) Aroclors by capillary gas chromatography (GC) with electron capture detection and total Aroclor quantification using EPA SW-846 Method 8082A.

2. Summary of Method

2.1. Samples are extracted with a pesticide analytical grade solvent. The extracts are further processed by concentration and a series of clean-up procedures. The sample extracts are then analyzed by injecting onto a gas chromatographic system equipped with an electron capture detector.

2.2. The purpose of this SOP is to provide a detailed written document for quantification of PCBs as Aroclors according to SW-846 Method 8082A specification.

2.3. This SOP provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs as Aroclors by gas chromatography. Sample extraction and cleanup procedures are described separately in additional laboratory Standard Operating Procedures.

2.4. Extensive knowledge of this SOP and EPA Method 8082A is required. The analysis portion of this method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analysis of PCBs by Method 8082A.

3.2. **Parameters**: The following PCB Aroclors can be determined by this method:

Compound	CAS Number
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

4. Applicable Matrices

4.1. This SOP is applicable in the determination and quantification of PCBs as Aroclors as outlined in EPA SW-846 Method 8082A. It is applicable to the following matrices: water, soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue samples.

5. Limits of Detection and Quantitation

5.1. The following are default reporting limits based on the lowest calibration standard:

Matrix	Sample Mass/Volume Extracted	Calibration Curve Low Standard	Extract Volume	RL (PQL) (all Aroclors)
Soil/Sediment Solid	10g	20ng/mL	25mL	0.050mg/kg
Water	1 Liter	5ng/mL	10mL	0.050ug/L
Water	1 Liter	5ng/mL	5mL	0.025ug/L
Biota	10g (wet weight basis)	20ng/mL	25mL	0.050mg/kg
Waste Oil	0.5g	20ng/mL	25mL	1.00mg/kg
Wipe	1 Wipe	20ng/mL	25mL	0.500ug/wipe

5.2. Individual MDLs are determined based on the Method Detection Limit (MDL) procedure outlined in 40 CFR Part 136, Appendix B. MDLs must be determined again whenever a major change in instrumentation or extraction methodology occurs.

5.3. MDLs are verified annually by the extraction and analysis of a low level MDL verification check sample. The Aroclor must be observed qualitatively in the MDL verification check sample.

5.4. Global MDL values can be obtained by request from the QA Department.

6. Interferences

6.1. Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters exhibit response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Laboratory method blanks must be thoroughly reviewed for presence of non-target peaks and comparison of samples with blank chromatographic patterns.

6.2. Elemental sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through the use of Method 3660.

6.3. Polychloroterphenyls (PCTs), polybrominatedbiphenyls (PBB), polychlorinated naphthalenes (PCN), as well as dioxins can co-elute with PCBs. Carry-over from these compounds, when in high concentration, is common if clean-up procedures are not followed. These materials may be removed through the use of specified clean-up procedures.

6.4. Pesticides can be a source of contamination through breakdown into components such as hexachlorobenzene (HCB). This chlorinated compound can carry-over on the GC column, and contaminate samples. Specified clean-up procedures should be followed to eliminate this as a source of contamination when analyzing PCBs. High concentrations of pesticides can cause carry-over on GC columns.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Sample Collection and Preservation:

7.1.1. Routine soil, sediment, sludge, solid, caulk, and concentrated liquid samples should be collected in 8 oz clear glass wide-mouth jars, fitted with a Teflon-lined cap. Aqueous samples should be collected in 1 liter amber glass bottles with a Teflon-lined cap. Project specific protocols may require that containers be pre-cleaned to EPA specification protocol A, “Specifications and Guidance for Contaminant-Free Sample Containers” (EPA 540/R-93/051). Protect samples from light.

7.1.2. All samples must be placed on ice or refrigerated at >0-6°C from the time they are collected until delivery to the lab. Samples that are collected within driving distance of the laboratory and delivered the same day may not have reached temperature acceptance limits. These samples are deemed acceptable if evidence of cooling is present (i.e., they are received with ice in the cooler).

7.2. Sample Shipment:

7.2.1. Sample Shipment is accomplished through a carrier such as Federal Express or United Postal Service for overnight 1-day delivery to the lab. Shipment is normally handled by the field personnel collecting the samples and coordinated with sample receiving department at the lab. Samples can also be picked up by the lab courier service if samples are collected within driving distance to the lab.

7.3. Sample Storage:

7.3.1. The samples must be protected from light and refrigerated at >0-6°C from time of receipt until they are removed from storage for extraction. Remaining sample material will be stored protected from light and refrigerated at >0-6°C. Sample will be disposed of or stored / archived according to project specifications.

7.3.2. Routine soil, sediment, sludge, solid, liquid and concentrated liquid samples are stored in a refrigerator dedicated for this type of sample.

7.4. Sample Extract Storage:

7.4.1. Sample extracts must be protected from light and refrigerated at >0-6°C during the analysis. After analysis is complete, sample extracts will be discarded after 60 days or can be archived in a freezer at less than -20°C for longer periods of time depending on the program requirements.

7.4.2. Field samples, sample extracts, and calibration standards must be stored separately.

7.5. Required Hold Time:

7.5.1. Extraction of solid samples by appropriate technique must be completed within one year from sample collection. Extraction of aqueous samples by appropriate technique must be completed within one year from sample collection. Sample extracts must be analyzed within forty days from extraction.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. Accuracy – The nearness of a result or the mean of a set to the true value. Accuracy is assessed by analysis of references samples and percent recoveries.

8.3. Analytical Batch –The basic unit for analytical quality control is the analytical batch, which is defined as samples which are analyzed together with the sample method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar matrices (e.g. water, sediment, soil, etc.).

8.4. Blank – A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix, however, a universal blank matrix does not exist for solid samples, but sometimes sodium sulfate is used as a blank matrix. The blank is taken through the appropriate steps of the process. A reagent blank is an aliquot of analyte-free water or solvent analyzed with the analytical batch. Field blanks are aliquots of analyte-free water or solvents brought to the field in sealed containers and transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two specific types of field blanks. Trip blanks are not opened in the field. They are a

check on sample contamination originating from sample transport, shipping and from site conditions. Equipment blanks are opened in the field and the contents are poured appropriately over or through the sample collection device, collected in a sample container, returned to the laboratory as a sample. Equipment blanks are a check on sampling device cleanliness.

8.5. Continuing Calibration Check Standard (CCCS) –The continuing calibration check standard contains all target analytes found in the calibration standards and is used to verify that the initial calibration is prepared correctly and that the instrument system is correctly calibrated. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.

8.6. Calibration Standard (ICAL)– A series of known standard solutions used by the analyst for instrument calibration. Calibration standards are prepared from primary standard and/or stock standard solutions.

8.7. CAS Number – An assigned number used to identify a chemical. CAS stands for Chemical Abstracts Service, an organization that indexes information published in Chemical Abstracts by the American Chemical Society and that provides index guides by which information about particular substances may be located in the abstracts. Sequentially assigned CAS numbers identify specific chemicals, except when followed by an asterisk (*) which signifies a compound (often naturally occurring) of variable composition. The numbers have no chemical significance. The CAS number is a concise, unique means of material identification. (Chemical Abstracts Service, Division of American Chemical Society, Box 3012, Columbus, OH 43210: [614] 447-3600).

8.8. Duplicate– A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

8.9. 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 as requested or required.

8.10. Initial Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical detector or method.

8.11. Instrument Calibration – Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

8.12. Laboratory Control Sample (LCS) – Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of an aliquot or reagent water or other blank matrix to which known quantities of the method analytes are added. The LCS is extracted and analyzed exactly like a field sample, and its purpose is to determine whether the analysis is in control and whether the laboratory is capable of making accurate and precise measurements.

8.13. Laboratory Method Blank – An analytical control consisting of all reagents and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

8.14. Matrix – The predominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

8.15. Matrix Spike – Aliquot of sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

8.16. Matrix Spike Duplicate – A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

8.17. Method Detection Limit (MDL) – The minimum constituent concentration that can be measured and reported with 99% confidence that the signal produced is different from the blank in a given matrix. The MDL is determined per 40 CFR Part 136 from a minimum of seven or eight replicate samples, taken through the entire preparation and analysis procedure. The standard deviation, *s*, of those replicates is multiplied by a student's t factor in order to calculate the MDL.

8.18. SDS – Safety Data Sheet. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs.

8.19. PCB- Polychlorinated biphenyls are a class of 209 individual chemical compounds (congeners), in which one to ten chlorine atoms are attached to biphenyl. Use of PCBs has made them a frequent environmental pollutant.

8.20. Precision – The agreement between a set of replicate measurements without assumption of knowledge of the true value. Precision is assessed by means of duplicate/replicate sample analysis.

8.21. Quality Control – Set of measures within a sample analysis methodology to assure that the process is in control.

8.22. Standard Curve – A standard curve is a curve which plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts which cover the expected range of the samples being analyzed. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

8.23. Stock Solution – Standard solution which can be diluted to derive the other standards.

8.24. Surrogate – Organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, calibration and check standards, samples (including duplicates and QC reference sample) and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

8.25. Surrogate Standard – A pure compound added to a sample in the laboratory just before processing so that the overall efficiency of a method can be determined.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Pace Analytical Services will use a Varian/Bruker Models 3800 and 450 (or equivalent) gas chromatograph (or equivalent), equipped with a Model 1177 split/splitless injector (or equivalent), temperature programmable oven, LEAP GC pal automatic sampler (or equivalent), and electron capture detector (or equivalent). A data system and integration of detector signal is interfaced to the gas chromatograph.

9.2. Chromatographic Data System: A data system for measuring peak height and peak area. An Empower computer network based workstation (Waters Corporation), will be employed to capture detector response and digitally store the chromatographic, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

9.3. Column (Primary Hydrogen Carrier Gas): ZB-1MS, Phenomenex Cat. No 7FD-G011-08; 20m x 0.18mm x 0.18um.

9.4. Column (Secondary Hydrogen Carrier Gas): ZB-5, Phenomenex Cat. No 7FD-G002-08; 20m x 0.18mm x 0.18um.

9.5. Hamilton Gastight syringes: 0.010-2.50mL

9.6. Class A pipettes: 1.00-100.0mL

9.7. Class A volumetric flasks: 5.0-100mL.

9.8. 8-dram vials and 4-dram vials for sample extract storage.

9.9. Pasteur pipettes.

9.10. 250mL and 100mL beakers, glass.

9.11. Disposable 1.0, 5.0, and 10.0mL pipettes.

9.12. Hexane, Burdick and Jackson-Pesticide Grade (or equivalent).

9.13. Acetone, Burdick and Jackson.-Pesticide Grade (or equivalent).

9.14. Toluene, Baker, (Cat.No. 9336-03) (or equivalent).

9.15. Methylene Chloride, Burdick and Jackson (Cat. No. 300-4) (or equivalent).

9.16. Ferrules: 0.4mm graphite/vespel, Restek 20229, and ¼" graphite ferrules, Restek 20210 or equivalent.

9.17. Injector septa: Thermolite Septa, Restek 20365 or equivalent.

9.18. Injector liner: Low Pressure Drop Liner w/Glass Wool, Restek 21033 or equivalent.

9.19. SGE Injector Syringe 10.0µL: SGE 002987 or equivalent.

9.20. Auto sampler vials: Snap vial 12x32mm Clear w/P, Microliter 11-5200 (or equivalent).

9.21. Snap Caps: 11mm Natural Snap Cap PTFE, Microliter 11-0051N-B (or equivalent).

10. Reagents and Standards

10.1. Aroclor Stock Standard Solutions:

10.1.1. Polychlorinated Biphenyls - Stock standards are prepared from individual Aroclor stock solutions from Accustandard. See Attachment 1 Table 1 for the exact preparation of each compound. Purchased stock standards should be stored according to manufacturer recommendations.

10.1.2. The stock standards are transferred into screw-cap Boston bottles and stored in a freezer 0°C, protected from light. Stock standards should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced after one year, or sooner if a problem with instrument calibration is detected.

PCB Formulation	Supplier	Catalog # (or equivalent)	Conc. (PPM)
A1016	Accustandard	C-216S-H-100x	10000.0
A1221	Accustandard	C-221S-H-100x	10000.0
A1232	Accustandard	C-232S-H-100x	10000.0
A1242	Accustandard	C-242S-H-100x	10000.0
A1248	Accustandard	C-248S-H-100x	10000.0

PCB Formulation	Supplier	Catalog # (or equivalent)	Conc. (PPM)
A1254	Accustandard	C-254S-H-100x	10000.0
A1260	Accustandard	C-260S-H-100x	10000.0
A1262	Accustandard	C-262S-H-10x	1000.0
A1268	Accustandard	C-268S-H-10x	1000.0
TCMX/DCBP (surrogate)	Ultra Scientific	CUS-4911	500/5000

*unless otherwise noted acetone is the solution used to make all stock standards.

10.2. Calibration Standards:

10.2.1. Calibration standards are prepared at five concentration levels using a prepared working standard. See Attachment I, Table 2 and for the preparation and exact concentrations of the working standards. The following five standards make up the initial calibration curve standard set for a High Level curve: 20ng/mL, 100ng/mL, 250ng/mL, 500ng/mL, 1000ng/mL. The following five standards make up the initial calibration curve set for a Low Level curve: 5ng/mL, 10ng/mL, 20ng/mL, 50ng/mL, 100ng/mL.

10.2.2. The two surrogates Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in the A1254 calibration standards. The standard for TCMX/DCBP is prepared by diluting 1mL of TCMX/DCBP custom standard solution (ULTRA, cat.#CUS-4911, at 500/5000 ng/mL) into a 1000mL volumetric flask resulting in a solution of TCMX/DCBP at 0.5/5.0ug/mL.

10.2.3. Refer to Attachment I, Table 3 for instructions on preparation of the calibration standards containing A1254 and the surrogates. Refer to Attachment 1, Table 2 for instructions on preparing the remaining calibration standards.

10.2.4. Transfer all calibration standards to ASE vials and store in a refrigerator at 0-6°C, protected from light. Calibration standards must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

10.3. PCB Continuing Calibration Stock Standards:

10.3.1. The stock standards are transferred into a screw cap boston bottles and stored in a refrigerator protected from light. Stock standard should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced annually, or sooner if a problem with instrument calibration is detected.

PCB	Supplier	Catalog #	Conc. (ug/mL)
A1016	Chem Service	S-11086J	1000
A1221	Chem Service	S-11087J	1000
A1232	Chem Service	S-11088J	1000
A1242	Chem Service	S-11089J	1000
A1248	Chem Service	S-11090J	1000
A1254	Chem Service	S-11091J	1000
A1260	Chem Service	S-11092J	1000
A1262	Ultra Scientific	EPA-1372	1000
A1268	Ultra Scientific	EPA-1382	1000

10.4. Continuing Calibration Standards:

10.4.1. The surrogate compounds Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in all Continuing Calibration Check Standards at a concentration near the mid-point of the surrogate calibration curve sequence. All continuing calibration standards are prepared independently from calibration standards, by using an alternate source purchased from standard vendors. Refer to Attachment II, Tables 1-3 for instructions on preparation of these standards.

10.5. Surrogated Instrument Blank Solution: A surrogated instrument blank standard is analyzed after each CCCS to check for instrument carry over. The surrogated instrument blank is prepared as follows:

10.5.1. High Level Instrument Blank: 2.0mL of the 0.5/5.0PPM TCMX/DCBP solution is diluted to 100mL in a volumetric flask set to volume in hexane. This results in a 10/100PPB TCMX/DCBP solution.

10.5.2. Low Level Instrument Blank: 1.0mL of the 0.5/5.0PPM TCMX/DCBP solution is diluted to 100mL in a volumetric flask set to volume in hexane. This results in a 5/50PPB TCMX/DCBP solution.

11. Calibration and Standardization

11.1. Gas chromatographic operation parameters: See Attachment III.

11.2. Initial GC Calibration:

11.2.1. GC calibration is performed by the external standard calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

11.2.2. Establish the gas chromatographic operating parameters outlined in the Procedure section and prepare the calibration standards at the five concentrations outlined in the Reagent and Standard section. Inject each calibration standard using the GC Autosampler and the parameters outlined in the Procedure section. Note: The same parameters are used for actual samples.

11.2.3. For each Aroclor, 5 peaks are selected to prepare calibration curves. The peaks selected from the multi-component Aroclor formulations were based on maximizing the separation for each Aroclor (i.e., minimizing peak overlap in retention time). Consideration was also given to selecting peaks that normally did not have problems with co-elution with interfering peaks or possible co-elution with organochlorine pesticides. The determined area of the five peaks selected for calibration is processed by the data workstation as a group, combining the area for calculations of the calibration factors. The following table lists the Aroclors that are included in the initial calibration and the peak numbers used.

Aroclor	**Peak Numbers**
A1016	6, 7, 8, 9, 10
A1221	1, 2, 3, 4, 5
A1232	5, 7, 8, 9, 10
A1242	6, 7, 8, 9, 10
A1248	11, 12, 13, 14, 15
A1254	16, 17, 18, 19, 20
A1260	20, 21, 22, 23, 24
A1262	20, 21, 22, 23, 24
A1268	23, 24, 25, 26, 27

11.2.4. For the initial calibration curve to be considered valid, the percent relative standard deviation of response factors must be less than 20% over the working range if average calibration factor quantitation is used. Note: the % RSD is a useful check for linearity through the origin and is used as a data quality indicator. In general an inverse weighted linear calibration curve with intercept is used for quantitation and is not replaced with the average calibration factor. For linear calibration curve the Correlation Coefficient R must be greater than 0.995.

11.2.5. Once linear calibration has been established it is subjected to an additional check. This check is the comparison of the calculated amount of the low calibration standard for each Aroclor against the expected amount of the standard using the % difference. Re-fitting the calibration data back to the model or calculating the % difference is determined by using the following equation:

% Difference = (Cc-Ce/Ce)x100

Where Cc=Calculated amount of standard, in mass or concentration units.

Ce=Expected amount of standard, in mass or concentration units.

The absolute value of the percent difference between these two amounts for

Every calibration level should be less than or equal to 20%.

11.2.6. Our laboratory uses a computer based chromatography software module (Water Corporation, Empower software) interfaced to the gas chromatograph. The workstation processes the detector signal, performs an analog to digital conversion, and stores the digitized chromatograms on the computer hard disk. Integration of peak areas and production of chromatograms is performed in the Empower software. All data analysis will be carried out in Empower including calculating calibration curves/response factors, report generation, and archival of data.

11.2.7. If a re-calibration is performed, the CCCS must be analyzed again and values calculated using the new relative response factors. If the CCCS fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (*i.e.,* GC gas leak, autosampler syringe plugged, broken injector liner).

11.3. Retention Time Windows:

11.3.1. The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Select a calibration standard and inject three times over a 72-hour time period.

11.3.2. For each peak calculate the standard deviation resulting from the variation in the three retention times for that peak.

11.3.3. The retention time window is defined as plus or minus three times the standard deviation of the three retention time determinations.

11.3.4. If the standard deviation of the selected peak is zero, then a default standard deviation of 0.01 minutes is used.

12. Procedure

12.1. Sample Extraction and Preparation: The following SOP's detail sample extraction procedures that are utilized in preparing samples for analysis by this analytical method:

SOP NAME	TITLE	EPA Method
S-NY-O-017	Extraction and Percent Lipids for Fish and Biota	8082A
S-NY-O-088	PCB Extraction Of Wipe	8082A, 3540C
S-NY-O-111	Waste Dilution EPA 3580 for PCB 8082	8082A, 3580A
S-NY-O-140	PCB Screening by GC	3510C, 3520C, 3545A
S-NY-O-141	SW-846 3510C H2O PCB extraction	8082A, 608, 3510C

12.2. Gas Chromatographic Procedures:

12.2.1. Prescreening of sample extracts: See standard operating procedure S-NY-O-140 for details on the PCB screening procedures used prior to final analysis by this method. Prescreening is a fast and effective way to determine if re-extracts are required and dilutions for over ranged samples. The GC will be standardized by using Aroclor 1221, Aroclor 1242, and Aroclor 1260. These three Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amount for final dilution for this determinative method. A three level calibration curve is utilized (0.50ug/mL, 2.5ug/mL, and 5.0ug/mL standards). The concentration of each Aroclor (grouped as Aroclor 1221, Aroclor 1242, and Aroclor 1260 only) in a sample will be calculated based on the extract volume (not the sample weight or volume) to supply solution concentration values that show if the extract needs to be diluted for final capillary GC analysis. If a dilution is necessary, sample extracts are diluted to a solution concentration near 0.500ug/mL, so ensuring each sample quantifies in the middle of the calibration curve.

12.2.2. Approximately 1.0mL of the final dilution extract is then transferred into a labeled autosampler vial.

12.2.3. The sequence of the analytical queue is set up in the Laboratory Information Management System (LIMS) as a unique batch file. This file contains the exact order in which standards, instrument blanks, and samples will be analyzed. Once the sample set is uploaded into the Empower acquisition/run screen and saved, the sample set is printed and the samples are loaded into the GC autosampler tray in the order specified by the sample set queue.

12.2.4. The following labeling will be used on the autosampler vial and for the sample set file created for the analytical queue.

12.2.4.1. The initial calibration standard will be labeled as 040516A, 040516B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

12.2.4.2. The instrument blanks will be labeled 070405B01, B02, B03, etc. Substitute the actual date of analysis in the file name.

12.2.4.3. The continuing calibration check standards will be labeled CS160405A CS160405B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

12.2.4.4. Samples are labeled with the laboratory identification number on the autosampler vial. In the sample set file the laboratory identification number, along with the client identification, sample weight, set volume and dilution are entered.

12.2.5. At this point the chromatography software can be initiated to start data collection. The gas chromatograph is placed into run mode and sample analysis is performed until the analytical queue is complete.

12.2.6. Peak Identification:

12.2.6.1. Target peaks are identified in unknown samples based upon Retention Time (RT). The retention time of an unknown peak must fall within the retention time windows established.

12.2.6.2. Besides using retention time windows to assign peak IDs, the analyst should also rely on their own experience in recognition of multi-response PCB chromatograms. Caution should be exercised when identifying peaks which elute near interferences present in samples and blanks. Comparison of sample chromatograms with method blank and field blank chromatograms is useful in determining chromatographic interferences.

12.2.6.3. This method should be applied with caution when used in determining PCB of interest in unknown sample for which no prior historical information exists. In this case confirmatory column analysis or confirmation by GC/MS analysis may be advised.

12.3. Data Reduction/Reporting:

12.3.1. Final peak assignments and quantitation calculations are performed within the software along with the current instrument calibration. The final concentration results are provided in the reporting section of the software. Final concentration results are reviewed by QA department or other approved manager before release to the client.

12.3.2. Data Qualifiers: Sample Concentration Reports (Certificates of Analysis, Data Package Form 1's and Electronic Data Deliverables (EDDs) are generated using the appropriate data qualifiers as defined in Laboratory Form F-NY-Q-033 "Report Definitions Page" current revision.

13. Quality Control

13.1. This section outlines the necessary quality control samples that need to be generated at the time of sample extraction. The results of the quality control measurement samples document the quality of the data generated.

13.2. Method Blank- With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples and organic-free reagent water blank is processed.

13.2.1. The method blank must exhibit PCB levels less than the matrix defined method detection limit (MDL) / reporting limit (RL). If the method blank exhibits PCB contamination above the reportable MDL/RL, the samples associated with the contaminated blank should be re-extracted and analysis repeated. If there is no original sample available for re-extraction, then the results should be flagged with a "B" indicating blank contamination. The value measured in the blank is reported for those samples associated with the particular blank out of criteria.

13.2.2. Method blanks can be reported with a "B" flag if the analyte detected is less than 10% of the regulatory limit associated with an analyte or is less than 10% of the sample result for the same analyte, whichever is greater.

13.3. Laboratory Control Sample (LCS)-

13.3.1. A Laboratory Control Sample (LCS), also referred to as a QC reference check standard, is extracted with each batch of samples at a rate of one per 20 samples. For water sample, spike one liter of laboratory organic free water, extract and analyze. For solid and tissue samples spike 10g of sodium sulfate, extract and analyze. For oil samples, spike 0.5g of PCB free oil, extract and analyze. An Aroclor is chosen for the LCS analyte, typically based on program requirements or expected sample contamination. Calculate the percent recovery for the PCB spike. If the percent recovery for the LCS is out of criteria, (70-130% or project-specified criteria) the analysis is out of the control and the problem should be immediately corrected.

13.3.2. The following are default Laboratory Control Sample Concentrations:

13.3.2.1. Aqueous Samples: 1.0mL of A1242 at 0.5ug/mL yielding a final sample concentration of 0.500ug/L.

13.3.2.2. Solid Samples: 1.0mL of A1242 at 12.5ug/mL yielding a final sample concentration of 1.25ug/g.

13.3.2.3. Note: Alternate spike concentrations and selection of Aroclors may be applicable based on project specific requirements.

13.4. Duplicate Analysis- Duplicate analysis of the same sample is performed to assess method precision. A duplicate can also be performed as a blind duplicate, so that identification with original sample is withheld. The analysis of a duplicate sample precludes that PCBs are to be found at appreciable levels in samples. If this is not known the analysis of matrix spike/matrix spike duplicates provide more consistent quality control information. The relative percent difference of the two measurements on the sample is calculated on total PCB concentration by the following equation:

RPD = (DUP1-DUP2)/AVG x 100

RPD = Relative Percent Difference
DUP1 = The greater of the measured values
DUP2 = The lesser of the measured values
AVG = Average of the two analyses
relative percent difference must be less than or equal to 30%

13.5. Matrix Spike and Matrix Spike Duplicate (MS/MSD):

13.5.1. A matrix spike is to be analyzed at a rate of one matrix spike per every 20 samples. Also matrix spike duplicate or duplicate sample is to be analyzed at a rate of one per every 20 samples. A matrix spike and an unspiked field sample/sample duplicate s may be appropriate in place of matrix spike/matrix duplicate, for soil and waste samples, where detectable amounts of organics are present.

13.5.2. The following are default Laboratory Matrix Spike Concentrations:

13.5.2.1. Aqueous Samples: 1.0mL of A1242 at 0.5ug/mL yielding a final sample added concentration of 0.500ug/L.

13.5.2.2. Solid Samples: 1.00mL of A1242 at 100ug/mL yielding a final sample added concentration of 10ug/g.

13.5.2.3. Note: Alternate spike concentrations and selection of Aroclors may be applicable based on project specific requirements.

13.5.3. Analyze one unspiked and one spiked sample. Calculate the percent recovery based on PCB concentration of both samples as follows:

P = A-B/T x 100

P = Percent recovery, %
A = concentration of analyte (PCB) in the spike sample aliquot
T = Know true values of the spike concentration
B = Background concentration of analyte (PCB) in the unspiked sample aliquot

13.5.4. Matrix spike recovery information is used to assess the long-term precision and accuracy of the method for each encountered matrix. Matrix spike/matrix spike duplicate results are not used alone to qualify an extraction batch. Generally, percent recovery for MS/MSD samples should be greater than or equal to 70% and less than or equal to 130% based on the total PCB concentration. If the percent recovery is outside the limits, all calculations should be checked and the data should be narrated to describe possible matrix interference.

13.6. Surrogates:

13.6.1. A surrogate compound is added to each sample, matrix spike, matrix spike duplicate, duplicate, method blank, and LCS at time of extraction. The surrogate compounds chosen for this method are Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP). The following are typical surrogate amounts added to normal encountered matrices. These amounts can be adjusted if the PCB background levels are high and the surrogate is being diluted out of analysis range.

13.6.1.1. Soil, sediment, sludge, oil, fuel oil, waste solvent, fish, other aquatic animals, tissue samples: 0.5mL of 0.5ug/mL TCMX/ 5.0ug/mL DCBP set to 25mL final extract volume.

13.6.1.2. Water: 1.0mL of 0.05ug/mL TCMX/ 0.5ug/mL DCBP set to 10mL final extract volume.

13.6.2. Surrogate compound is added to all instrument blanks that are analyzed after CCCS. Surrogate compound must meet lab established limits of TCMX 47-123% and DCBP 35-153%.

13.6.3. The surrogate recoveries must fall within lab established limits of TCMX 47-123% and DCBP 35-153%. If percent surrogate recovery is not within laboratory established limits for either surrogate, the following steps are required.

13.6.3.1 Review calculations that were used to generated surrogate percent recovery values to make certain there are no errors.

13.6.3.2 Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

13.6.3.3 Review data for chromatographic interferences.

13.6.3.4 Re-extraction and/or re-analysis of samples may be indicated if problems persist with surrogate recoveries. If the surrogate percent recovery is out of limits on the re-extracted samples, low or high surrogate recovery is due to matrix affects and the data can be reported as estimated. If

above steps do not lead to satisfactory results then consult with organics manager to resolve the situation.

13.7. Continuing Calibration Check Standard (CCCS):

13.7.1. The initial CCCS is from an alternative source independent of the calibration check standards. It is prepared at a concentration approximately equal to the midlevel calibration standard. This standard is analyzed after the initial calibration standards, every tenth injection, and at the end an analytical sequence. One check standards must be run within a 12 hour analytical shift. The percent recover must be ± 20% of the true value.

13.7.2. If the criterion is exceeded, the analyst should inspect the system to determine the cause and perform maintenance as necessary. The system can then be recalibrated and sample analysis can proceed. Note that all samples which are not bracketed by valid check standards must be re-analyzed when the system is in-control.

13.8. Retention Time:

13.8.1. The retention time (RT) windows are established from the continuing calibration check standard (CCS) peak retention times. The CCS is analyzed three times over a 72-hour period and the standard deviation is calculated from the three retention time measurements. The standard deviation is multiplied by three and this establishes the retention time window for each quantified peak (±3SD). Use the retention time for a peak in the continuing calibration check standard to determine the midpoint of the retention time window for the analysis sequence. If the continuing calibration checks fall outside of these windows update the windows using the previous check standard. If the retention times are still outside the established windows instrument maintenance must be performed and recalibration may be required.

13.8.2. This function is performed in the chromatography software graphically as vertical dropdown retention time markers with retention time window brackets. Besides using the retention time window to assign peaks for quantification, the analyst should also rely on their experience in pattern recognition of multi-response sample analysis.

13.8.3. Retention time studies are available upon request form the QA department.

13.9. Analytical Sequence Queue: The following is an example of the order that initial calibration standards, continuing calibration check standards, method blanks, QC samples, and samples are placed in an analytical sequence. A continuing calibration check standard is run after every ten samples in the analytical sequence. All analytical sequences must end with a continuing calibration check standard regardless of the number of samples. Below is an example of an analytical sequence.

Injections	Material Injected
1-2	Hexane Blank
3-47	Initial Calibration Standards
48	Hexane Blank
49-57	Continuing Calibration Check Standard
58	Hexane Blank w/surrogates
59-68	Samples analyses, including method blanks, matrix spikes, matrix duplicates, matrix spike duplicates, and QC reference check standard. A maximum of ten samples between continuing calibration check standards
69	Continuing calibration check standard
70	Hexane Blank w/surrogates
71-82	Another set of injections that are the same type as the 59-70 sequence

13.10. PCB Aroclor Qualitative Identification and Secondary GC Column Confirmation:

13.10.1. Positive identification of PCB Aroclors is based on comparison of retention time of the five selected quantitation peaks and major non-quantitation peaks for the unknown sample with retention time of reference standards (continuing calibration check standards). Additionally pattern recognition is used for comparison of unknown samples with reference standards for positive identification. Confirmation of Aroclor presence by secondary GC column analysis may be necessary for highly altered/degraded PCB patterns or for programs including PCB air monitoring, US-EPA CLP protocol and other projects as specified in the site sampling and analysis quality assurance plan.

13.10.2. In cases where multiple Aroclors are present with overlapping chromatographic patterns or interferences are encountered that are not removed with extract cleanup processes one or two quantitation peaks may be dropped and not used for quantitation. A minimum of 3 quantitation peaks must be used for all unknown samples and standards. When quantitation peaks are dropped for a sample or standard the corresponding peaks are also dropped in the initial calibration sequence for calculation purposes.

13.10.3. Dual Column/Confirmatory Column Analysis by GC- Inject samples under same operating conditions and analytical run QA/QC parameters on a secondary GC column of dissimilar phase (e.g., ZB-1 and ZB-5). Note: If using dual GC column system, samples are injected sequentially through separate injection ports onto both columns. Samples are analyzed and concentration results are reported.

13.10.4. Dual Column/Confirmatory Column Laboratory Default by SW-846:

13.10.4.1 Report lowest concentration of the 2 column results for each individual Aroclor on the merged EDD, Form 1 or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects).

13.10.4.2 If **RPD percent** exceeds 40% report the lowest concentration result of the two analyses unless observed chromatographic interference or instrumental analysis QA/QC indicates the higher value may be more accurate. P-flag all excursions > 40% and describe interferences or rationale for reporting lower value in Data Narrative.

13.10.4.3 If a concentration is above the PQL on one column and below the PQL on the second column, the qualitative presence is not confirmed and the sample is reported as not detected. **Note: If reporting to the MDL is required do the following:** For reporting to the MDL: a) If one result is

greater than the PQL and other result is < PQL (J-flag) Report the **highest** result as confirmed (*unless interference or QC reasons indicate lower value*); b) If one result is above MDL (J-Flag) and second is Not Detected report the concentration as **not detected**. (Presence not confirmed); c) If both results are J-Flag values (< PQL) report the lowest value of the two.

13.10.5. **USEPA-CLP/ASP Program Protocols:**

13.10.5.1 Report **Lowest** Value of the 2 column results for each individual Aroclor on the merged EDD, Form 1 or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects. E.g. Air Monitoring for EPA TO-10A alternative reporting may be based upon total PCB values for PCB- Total regulated projects).

13.10.5.2 If **Percent Difference** (not RPD%) exceeds 25% then P-flag all excursions > 25%. Note any chromatographic interferences present in Case Narrative.

13.10.5.3 If one result is greater than PQL and other result is < PQL (J-Flag) Report the **lowest** result (J-Flagged) value (*confirmed hit*).

13.10.5.4 If one result is above MDL (J-Flag) and second is Not detected, report the concentration as **not detected** (*presence not confirmed*).

14. Data Analysis and Calculations

14.1. PCB Solution concentration calculation from initial Calibration by Linear Regression:

Yi= aXi + b

Xi = Calibrated Solution Concentration (ng/mL)
Yi = total area response of 5 PCB quant. peaks (uV-Sec.)
a = slope
b = intercept

Note: In those instances where samples may be quantitated with 3-4 peaks due to interference or overlap, the Empower system automatically quantitates against the calibration using only the area of the selected peaks.

Unknown Solution Conc. X = (Y –b) a

Y = Total area response of PCB Chromatogram (uV-Sec.)
a = slope of ICAL by linear regression
b = intercept of ICAL by linear regression

14.2. Capillary GC: Sample calculations:

14.2.1. The concentration of each identified PCB Aroclor in a sample will be calculated based on the sample weight or volume.

14.2.2. The PCB solution concentration of the extract is calculated as follows:

Solution Conc. = (Y –b)/a

Y = Total area response of PCB Chromatogram (uV-Sec.)
a = slope of ICAL by linear regression
b = intercept of ICAL by linear regression

14.3. Final concentration of samples- Calculations of final PCB concentrations will vary upon matrix, calculations are as follows:

14.3.1. **Soil/Sediment/Solids**:

Final Conc. = (Sol. Conc.) * (V)*DF/ (M)* (%Total Solids) (1/1000) ug/g

Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

14.3.2. **Water:**

Final Conc.= (Sol. Conc.) * V*DF/[(Vt)](1/1000) ug/L

Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
Vt= Total Volume Extraction (L)

14.3.3. **Biota Tissue:**

Final Conc. = (Sol. Conc.) * (V)*DF/(M)(1/1000) ug/g

Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

14.3.4. **Waste Oil**:

Final Conc. = (Sol. Conc.) * (V)*DF/ (M)*(%Total Solids) (1/1000) ug/g

Sol Conc. = Solution Concentration (ng/mL)
V = concentrated extract volume (mL)
DF = analytical dilution factor
M = mass extracted (g)

14.4. The calculated concentration for each PCB Aroclor will be compared to its respective sample-specific reporting limit (RL) and method detection limit (MDL). The results with concentrations at or above the MDL but below RL will be reported as detects and flagged as estimated J. The results for peaks with concentrations at or above the RL would be reported as unqualified numeric values.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. The GC analyst is responsible for generating the data and also is the initial individual to review the data. This would include inspection of the chromatographic data, processing the raw data, producing all required data forms, inspection of calibration curves for compliance, surrogate recovery, laboratory control spike recovery, matrix spike/matrix spike duplicate recovery, and continuing calibration compliance.

15.2. Once the initial review of the data is performed by the analyst, decisions are made at that time to accept the data if all criteria are met or to reject sample data if any of the quality control parameters or limits are out of control. Depending on the situation, samples requiring re-extraction will be notified to the appropriate extraction personnel, sample extracts requiring re-injection will be queued for analysis, new calibrations may have to be performed, or samples re-analyzed due to failing continuing check standards.

15.3. The analyst may also consult with the Quality Manager as to the best form of action to take or if the situation warrants corrective action beyond routine practices. If no recourse is available and the data is to be reported out of criteria, a Case Narrative Report is generated and the deviation is documented and reported to the client. The Case Narrative Report is filed with the data and is also useful for production of case narratives that are issued with the final data reports. If a problem exists that requires follow-up to rectify, a LabTrack Ticket (LTT) is issued to document the problem found, steps taken to resolve the problem, and what samples were affected. This LTT is reviewed by the Quality Manager and lab management to verify that appropriate actions have been taken to correct the problem.

15.4. Please see Table 16.1 below for specific Quality Assurance Acceptance Criteria.

16. Corrective Actions for Out-of-Control Data

16.1. The table below outlines the data assessment, acceptance criteria, and corrective action procedures for out-of-control data.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	The five point calibration is analyzed initially for all Aroclors and when Continuing Calibration Check standard fail criteria.	- %RSD≤20% for the relative response factors for the calibration standards if using average response factor calibration. - Correlation Coefficient R must be >0.995 for Linear Regression.	- Re-analyze the initial calibration standard and/or evaluate/correct instrument malfunction to obtain initial calibration and continuing calibration check standards that meet criteria.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Check Standard (CCCS)	- Initially analyze a CCCS immediately following an initial calibration. - After the initial CCCS of the sequence, a CCCS must be analyzed after 10 samples. - Analytical sequence must end with analysis of a CCCS. - CCCS must be analyzed at least once per 12 hour analytical shift. - CCCSs are rotated through all Aroclors in order.	- Calibration factor for the continuing calibration check must ±20% of the true value. - Retention time of all quantitated peaks must be within RT window (reset with each initial CCCS of a sequence). - All samples must be bracketed by a CCCS that meet all criteria stated above.	- If the reason for the failure of the CCCS appears to be a poor injection (or a degraded standard solution), the CCCS will be re-injected (or re-prepared and re-injected) immediately following the failed CCCS. This can only occur if the instrument is being attended by an analyst. If upon re-injection, the CCCS meets all the acceptance criteria and there is no apparent impact on the sample data the analytical sequence will continue and samples will not be reanalyzed. The associated sample data will be reported. - If CCCS failure was not due to a poor injection (or degraded standard solution) or the instrument was unattended at the time of the CCCS failure, correct system, if necessary, and recalibrate. Initial calibration and CCS criteria must be met before sample analysis may begin. Samples that are not bracketed by complaint CCCSs must be reanalyzed. -If acceptable CCCSs are observed later in the sequence, samples bracketed by acceptable CCCSs will be reported. Samples between the failed CCCS and prior/ subsequent complaint CCCS will be re-analyzed.
Retention Time (RT)	- Use the retention time for peak in the CCSs to determine midpoint of the relative retention time window for the analysis sequence. -Each sample analysis: Rely on RT windows to identify PCB Aroclor to report. Also use pattern recognition and professional judgment for peaks that shift from RT windows, because compound composition may shift RT for GC peaks.	- Each quantitated peak and surrogate peak should be within established windows.	-Inspect chromatographic system for malfunction, correct problem. Perform re-analysis if necessary.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Method Blank	-One per extraction batch of ≤20 samples of the same matrix per day. -Should be analyzed with other associated batch QC samples on the same instrument, but not all samples. -Must undergo all sample preparative procedures.	- Concentration does not exceed the RL/MDL for any PCB Aroclor. - Must meet in house surrogate criteria of TCMX 47-123% and DCBP 35-153%.	- Re-analyze method blank to determine if instrument contamination was the cause. If method blank re-analysis passes, then report samples. -If method blank is found to contain PCB contamination above the RL/MDL for any PCB Aroclor compound, then re-extract and re-analyze all associated samples. -If no sample is available for re-extraction, report data B flagged to indicate method blank contamination.
Laboratory Control Sample (LCS)	- One per extraction batch of ≤20 samples per matrix per day. - Should be analyzed with other associated batch QC samples on the same instrument, but not all samples.	-Percent recovery must be within method limits. - Must meet Aroclor spike criteria of 70-130% recovery - Must meet in house surrogate criteria of TCMX 47-123% and DCBP 35-153%.	-Re-analyze LCS to determine if instrument was the cause. If LCS passes, then report samples. -If LCS recovery is still out of limits, the re-extract and re-analyze all associated samples. -If no sample is available for re-extraction, report data flagged to indicate LCS failed recovery.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	-One MS/MSD per extraction batch of ≤20 samples per matrix . - A MS and a sample/sample duplicate may be appropriate in place of MS/MSD matrices where detectable amounts of analytes are present.	- Percent recovery for MS must be within method limits - If MS/MSD is analyzed, relative percent difference (RPD) should be within 30%. - Must meet Aroclor spike criteria of 70-130% recovery - Must meet in house surrogate criteria of TCMX 47-123% and DCBP 35-153%. (unless original unspiked sample is also outside of criteria)	-Re-analyze MS and/or MSD to determine if instrument was the cause. If MS and/or MSD pass, then report samples. -Check for errors such as calculations and spike preparation. -Check original unspiked sample results and surrogate recovery for indications of matrix effects. -If no errors are found, and the associated LCS is within limits, then sample matrix effects are likely the cause. Note exceedance in case narrative.

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Surrogates	-Calibrated as target compound in the Aroclor A1254 standards. -Surrogates are added to all calibration check standards, blanks (including instrument blanks run after CCCS), samples and QC samples.	- Must meet in house surrogate criteria of TCMX 47-123% and DCBP 35-153%.	-Re-analyze the affected sample or QC sample to determine if instrument was the cause. If surrogate passes, then report samples. -Check for errors in surrogate calculation and surrogate solutions. -If no problem is found, then re-extract and re-analyze the sample. -If re-extraction is within limits and sample extract holding time, then report only the re-analysis. -If the re-extraction is within limits, but out of extraction holding time, then report both sets of data. -If the re-extraction produces surrogate recovery still out of limits, then report both sets of data. -If no sample exists for re-extraction, report data flagged to indicate surrogate failed recovery or have a client re-sample.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Data that is detected to be out-of-control for any reason, when compared to method acceptance criteria, will addressed in the following manner:

17.1.1. If the problem exists with the gas chromatographic instrumentation, appropriate action will be taken to repair and perform maintenance to bring the instrument back to operation condition. Once the instrumentation is determined to be correctly operating analysis can begin again.

17.1.2. If the problem exists with calibration standard solutions, the analyst will prepare new standards and discard the standard solutions that are suspect. Instrument calibration can be performed and analysis can begin once system is control.

17.1.3. If the problem exists with sample extraction and extract preparation, the extraction step that is producing the out of-control situation will be diagnosed and rectified. Once the troubleshooting procedures correct the problem extraction can once again occur and analysis can continue.

17.1.4. In situations where data is reported under out-of-control conditions, the data will be annotated with data qualifiers and/or appropriate descriptive comments defining the nature of the excursion in the sample case narrative. If warranted, a LabTrack Ticket will be issued to define the problem, steps to correct the problem, and final resolution.

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. Initial Demonstration of Capability (IDOC) Procedure:

18.2.1. Prepare 4 replicates of a fortified laboratory blank sample (using laboratory reagent water or sodium sulfate) by spiking each sample with 1.0mL of 0.500ug/mL Aroclor solution for water samples and 0.100mL of 100ug/mL Aroclor 1242 solution for solid samples. The Aroclor type used for spiking should be rotated on a yearly basis. Prepare one method blank sample with the batch.

18.2.2. For each replicate the recovery value of the sample must fall in the range of 70-130% (or established lab limits) and the percent RSD must be <20 % for the method performance to be considered acceptable.

18.2.3. This procedure must be repeated using four fresh samples until satisfactory performance has been demonstrated. The initial demonstration of capability is used primarily to preclude the laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it. It is expected that as laboratory personnel gain experience with this method the quality of data will improve beyond those required here.

18.3. Continuing Demonstration of Performance Procedure:

18.3.1. Annual continuing demonstration of performance may be satisfied by a repeat Initial Demonstration of Performance, the acceptable analysis of an unknown samples (for example PT test sample), or the acceptable analysis of 4 consecutive Laboratory Control Samples. Records of continuing demonstration of performance are maintained by the laboratory Quality Assurance Department.

18.3.2. With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples an organic-free reagent water blank is processed.

18.3.3. The method blank should exhibit PCB levels less than the practical quantification limit or reporting limit (PQL or RL). If the method blank exhibits PCB contamination above the reportable quantitation limit, the samples associated with the contaminated blank should be re-extracted and analysis repeated when appropriate. If there is no original sample available for re-extraction or if the associated sample concentrations greatly exceed the blank concentration, then all positive concentration results for the associated samples should be flagged with a "B" indicating blank contamination and a case narrative describing the situation prepared.

18.3.4. A matrix spike/ matrix spike duplicate is to be analyzed at a rate of 1 matrix spike/ matrix spike duplicate per every 20 samples. A duplicate sample may be prepared in lieu of a matrix spike duplicate when detectable PCB concentrations are known to be present.

18.4. Method Detection Limit:

18.4.1. A method detection limit will be determined for this method whenever major modification to the extraction or analysis procedures are made or at a minimum frequency of every 2 years. A minimum of seven laboratory organic free water samples or sodium sulfate will be prepared and spiked with chlorinated PCB methyl esters mixture, at a low level and taken through all extraction and analytical procedures.

$$MDL = S * t_{(n-1,\ 1-alpha=0.99)}$$

S = Standard deviation of the replicate analyses
n = Number of replicates
$t_{(n-1,\ 1-alpha=0.99)}$ = Student's t value for the 99% confidence level with n-1
For example: t for 8 replicates = $t_{(7,0.99)}$ = 2.998

18.4.2. The determined MDL must be less than the concentration spiked but greater than one tenth (1/10) the spiked concentration. If not, repeat the MDL determination at an appropriate spike concentration for affected analytes.

19. Method Modifications

19.1. SW-846 EPA Method 8082A specifies to use Aroclors 1016 and 1260 for matrix spike samples unless specified by the client. The laboratory uses Aroclor 1242 as the default spike analyte due to historic contamination in the area coming from this Aroclor.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Safety glasses and disposable gloves must be worn when handling samples and extracts.

22.2. All manipulations of sample extracts should be conducted inside a chemical fume hood. Manipulation of sample extracts outside of a fume hood should be minimized by the analyst.

22.3. Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize themselves with the precautions for handling solvents and chemicals used to process samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

22.4. Samples remaining after analysis should either be returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for assistance and also SOP S-NY-O-054, disposal of laboratory waste.

23. Waste Management

23.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste generated during the execution of this method.

23.2. Please refer to SOP S-NY-W-054 regarding how hazardous waste is handled and disposed of by the laboratory.

24. Pollution Prevention

24.1. Pollution prevention is practiced in the laboratory by minimizing usage of solvents and chemicals, so that disposal of waste generated is held to the smallest amount possible. This is directly linked to the types of extraction procedures in place at the laboratory to reduce the volumes of solvents used for semi-volatile extraction procedures. Pace Analytical Services employs extraction procedures such as continuous

liquid/liquid and solid phase extraction methods to reduce solvent requirements for water extraction protocols and ASE and Soxhlet extractions for solid matrices.

24.2. Pollution prevention also relies on minimizing to the best extent the chemicals and solvents required to perform extraction and analysis procedures. The laboratory personnel strive to purchase chemicals and standards that will be consumed based on anticipated workload. For additional information about laboratory pollution prevention, please refer to laboratory SOP S-NY-S-168.

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. EPA SW-846 Method 8082A "Test Methods for Evaluating Solid waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

25.5. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures of the Analysis of Pollutants", July, 1988.

25.6. Standard Methods for the Examination of Water and Waste Water", 19th Edition 1995, American Public Health Association, American Water Works Association, Water Pollution Control Federation.

25.7. New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual", Wadsworth Center for laboratories and Research, 1996.

25.8. "Guide to Environmental Analytical Methods", third edition, Genium Publishing Corporation, 1997.

25.9. "Specifications and Guidance for Contaminant-Free Sample Containers" (EPA 540/R-93/051), Office of Solid Waste and Response, December 1992.

25.10. Pace SOP S-NY-O-017 "Soxhlet Extraction of Fish and Biota Samples for PCBs- EPA 3540C".

25.11. Pace SOP S-NY-O-088 "Soxhlet Extraction of PCB (Wipes) for PCB analysis- EPA 3540C/8082A".

25.12. Pace SOP S-NY-O-111 "Waste Dilution Extraction and Clean-up for PCB samples- EPA 3580A/8082A".

25.13. Pace SOP S-NY-O-140 "Screening of PCBs by GC NEA Method".

25.14. Pace SOP S-NY-O-141 "Sep Funnel Extraction for PCB analysis- EPA 3510C/608/8082A".

25.15. Pace SOP S-NY-W-054 "Waste Handling and Management".

25.16. Pace SOP S-NY-S-168 "Pollution Prevention".

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: PCB Stock Standard/Calibration Standard Preparation.

26.2. Attachment II: Continuing Calibration Check Standard Preparation.

26.3. Attachment III: GC Operating Parameters.

26.4. Attachment IV: Chromatograms.

27. Revisions

Document Number	Reason for Change	Date
S-NY-O-314-rev.01	General: converted to new format. General: removed all references to air matrix (PUF). Sections 10.1-10.4: updated standards used and instructions for working standard creation Sections 11.2.5 and 13.6.2: added Section 12.1: revised to match current SOP list. Sections 13.6.3 and 16.1: updated surrogate recovery acceptance limits Section 16.1: specified that surrogate calibration is done with Aroclor 1254 Sections 25.1-25.3: added standard Pace references. Section 26.1: updated attachment	12Feb2015
S-NY-O-314-rev.02	Sections 13.10.2 and 14.1: added documentation regarding quantitation with 3-4 peaks	30March2015
S-NY-O-314-rev.03	Section 5.2: changed to reference 40 CFR MDL procedure Section 12.3.2: removed qualifiers and referred to Report Definitions Page controlled document form Sections 15.3 and 17.1.4: updated to reference LabTrack system Section 19.1: added documentation of modification for use of Aroclor 1242 as default spiking analyte	10June2015
S-NY-O-314-rev.03a	Sections 1, 18.3.4: edited text to eliminate redundancy Sections 7.1.1, 25: added EPA reference Sections 8.4, 13, 15.4, Table 16.1: fixed typos Section 8.17: added reference to 40 CFR Part 136 Section 9: deleted helium as carrier gas, added gastight syringes and Class A pipettes Section 10: clarified standard storage and use of equivalent standards and acetone for stock standards Section 10.5: added use of surrogated instrument blanks Section 11.2.4: updated initial calibration curve criteria Section 11.3.4: eliminated text that did not apply Section 13.9: corrected text to consistently specify 10 sample injections between CCCSs Section 25: added Pace internal SOPs as references	26Sept2016

Attachment I: PCB Stock Standard/Calibration Standard Preparation

Table 1: PCB Stock Standard Preparation Table

PCB Stock Standards	Init Volume (mL)	Final volume (mL)	Conc. (ppm)
A1016	5.0	50	10.0
A1221	5.0	50	10.0
A1232	5.0	50	10.0
A1242	5.0	50	10.0
A1248	5.0	50	10.0
A1254	5.0	50	10.0
A1260	5.0	50	10.0
A1262	1.0	100	10.0
A1268	1.0	100	10.0

Unless otherwise noted hexane is the solution used to make all dilutions. *Custom Order

Attachment I: PCB Stock Standard/Calibration Standard Preparation (continued)

Table 2: PCB Calibration Standard Preparation Table (High Level Calibration Curve)

Initial Volume (mL)	Initial Conc. (ug/mL)	Final Volume (mL)	Final Concentration (mg/L)							
			A1016	A1221	A1232	A1242	A1248	A1260	A1262	A1268
5.0	10.0	50.0	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
2.5	10.0	50.0	0.500	0.500	0.500	0.500	0.500	0.500	0.500	0.500
1.25	10.0	50.0	0.250	0.250	0.250	0.250	0.250	0.250	0.250	0.250
0.500	10.0	50.0	0.100	0.100	0.100	0.100	0.100	0.100	0.100	0.100
5.0	1.00	50.0	0.020	0.020	0.020	0.020	0.020	0.020	0.020	0.020

See Table 3 for A1254 Standard Preparation (high level)

Table 2A: PCB Calibration Standard Preparation Table (Low Level Calibration Curve)

Init. Volume (mL)	Initial Conc. (ug/ml)	Final Volume (mL)	Final Concentration (ppm)							
			A1016	A1221	A1232	A1242	A1248	A1260	A1262	A1268
0.5	10.0	50.0	0.100	0.100	0.100	0.100	0.100	0.100	0.100	0.100
2.5	1.0	50.0	0.050	0.050	0.050	0.050	0.050	0.050	0.050	0.050
1.0	1.0	50.0	0.020	0.020	0.020	0.020	0.020	0.020	0.020	0.020
1.0	0.500	50.0	0.010	0.010	0.010	0.010	0.010	0.010	0.010	0.010
0.50	0.500	50.0	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005

See Table 3A for A1254 Standard Preparation (low level)

Attachment I: PCB Stock Standard/Calibration Standard Preparation (continued)

Table 3: PCB A1254 Calibration Standard Preparation Table (for High Level Curve)

Initial Volume (mL) A1254	Initial Conc. (ug/mL) A1254	Initial Volume (mL) 0.5/5.0 -ppm Surrogate	Final Volume (mL)	Final Concentration (ppm)		
				A1254	TCMX	DCBP
5.0	10.0	0	50	1.000	0	0
2.5	10.0	0	50	0.500	0	0
5.0	10.0	2.00	50	1.000	0.020	0.200
2.5	10.0	1.00	50	0.500	0.010	0.100
1.25	10.0	0.800	50	0.250	0.008	0.080
0.500	10.0	0.500	50	0.100	0.005	0.050
1.00*	1.00	0.200	50	0.020	0.002	0.020

*This initial volume is of the A1254 1.00ug/mL secondary stock solution WITHOUT surrogates

Table 3A: PCB A1254, TCMX and DCBP Calibration Standard Preparation Table (for Low Level Curve)

Initial Volume A1254 (mL)	Initial Conc. A1254 (ug/mL)	Initial Volume (mL) 0.5/5.0 -ppm Surrogate	Final Volume (mL)	Final Concentration (ppm)		
				A1254	TCMX	DCBP
0.5	10.0	0.800	50	0.100	0.00800	0.0800
2.50	1.000	0.500	50	0.050	0.00500	0.0500
1.0	1.000	0.400	50	0.020	0.00400	0.0400
1.0	0.500	0.250	50	0.010	0.00250	0.0250
0.50	0.500	0.100	50	0.005	0.00100	0.0100

Attachment II: Continuing Calibration Check Standard Preparation

Table 1: PCB Continuing Calibration Working Standards prepared from 1000ug/mL Stock Standards

PCB		Initial Volume (mL)	Final Volume (mL)	Concentration (ppm)
A1016		1.0	100	10.0
A1221		1.0	100	10.0
A1232		1.0	100	10.0
A1242		1.0	100	10.0
A1248		1.0	100	10.0
A1254		1.0	100	10.0
A1260		1.0	100	10.0
A1262		1.0	100	10.0
A1268		1.0	100	10.0

Attachment II: Continuing Calibration Check Standard Preparation (continued)

Table 2: PCB Continuing Calibration Standards (High Level) prepared from 10ug/mL CCV Working Standards and all contain surrogates

PCB	Surr. Volume* (mL)	Initial Volume Aroclor (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (ppm)	Aroclor Concentration (ppm)
A1016	2.0	5.0	100	0.010/0.100	0.500
A1221	2.0	5.0	100	0.010/0.100	0.500
A1232	2.0	5.0	100	0.010/0.100	0.500
A1242	2.0	5.0	100	0.010/0.100	0.500
A1248	2.0	5.0	100	0.010/0.100	0.500
A1254	2.0	5.0	100	0.010/0.100	0.500
A1260	2.0	5.0	100	0.010/0.100	0.500
A1262	2.0	5.0	100	0.010/0.100	0.500
A1268	2.0	5.0	100	0.010/0.100	0.500

*Surrogate stock solution 0.500ug/mL TCMX and 5.0ug/mL DCBP

Attachment II: Continuing Calibration Check Standard Preparation (continued)

Table 3: PCB Continuing Calibration Standards (low Level) prepared from 10.0ug/mL CCV Working Standards and all contain surrogates.

PCB	Surr. Volume* (mL)	Initial Volume Aroclor (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (ppm)	Aroclor Concentration (ppm)
A1016	1.0	0.500	100	0.005/0.050	0.050
A1221	1.0	0.500	100	0.005/0.050	0.050
A1232	1.0	0.500	100	0.005/0.050	0.050
A1242	1.0	0.500	100	0.005/0.050	0.050
A1248	1.0	0.500	100	0.005/0.050	0.050
A1254	1.0	0.500	100	0.005/0.050	0.050
A1260	1.0	0.500	100	0.005/0.050	0.050
A1262	1.0	0.500	100	0.005/0.050	0.050
A1268	1.0	0.500	100	0.005/0.050	0.050

*Surrogate stock solution 0.500ug/mL TCMX and 5.0ug/mL DCBP

Attachment III: GC Operating Parameters

GC-10 8082 High Level Method - Hydrogen

GC #: 10
Method: Hydrogen 8082
GC Method #: 2
Date: 02/09/2012
Analyst: MTH
File Name:
Column: Front ZB-1 MS 20m 0.18 0.18
Middle ZB-5 20m 0.18 0.18

Sample Delivery: SEE LEAP PARAMETERS

Column Oven:

Step	Temp (°C)	Rate (°C/min)	Hold (min)	Total (min)
Initial	150		1.41	1.41
	290	17.5	0.65	10.04

Stabilization Time (min): 0.5

Injector: Front CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Flow/PSI(Front EFC, Type 1):
Carrier Gas Hydrogen

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	*30 **		10.00	10.00

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Injector: Middle CP-1177

1177 Oven Power: ON
1177 Temperature (°C) 300

Time	Split State	Split Ratio
Initial	ON	30

Flow/PSI(Front EFC, Type 1):

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	*30 **		10.00	10.00

Constant Flow Mode Enable: NO
Column Flow Rate (ml/min): 5.0

Detector:

	Front ECD	Middle ECD
ECD Oven Power:	ON	**ON**
Temperature (°C)	300	300
Electronics:	ON	ON
Range:	1	

Time	Range	Autozero
Initial	1	YES

Front ECD Adjustment		
Time Constant:	Fast	Fast
Cell Current:	CAP	CAP
Contact Potential (mV):	250 *	365 *
Date of last adjustment	01/23/2012	01/23/2012
Make-Up Flow (ml/min):	35.0	35.0

*values may change with use

Analog Output

Detectors:			
	Front: ECD	Attenuation	1
	Middle: ECD	Attenuation	1
	Rear: None		

Attachment III: GC Operating Parameters (continued)

Leap GC Pal Parameters					
Sample injection Methods					
Method	**GC Dual**	**GC Duals**		**Method**	**GC Inj s**
Cylcle	GC Dual	GC Dual		**Cylcle**	GC Inj S
Syringe	10uL	10uL		**Syringe**	10uL
1. Sample Vol	1.0uL	1.0uL		**1. Sample Vol**	1.0uL
1. Air Vol	1.0uL	1.0uL		**Solvent Plug**	200nL
1. Inject to	GC Inj 1	GC Inj 1		**Slv Source**	Standard
Inj Time Diff	0s	0s		**Int Standard**	0nL
2. Sample Offs	1	0		**Std Source**	Standard
2. Sample Vol	1.0uL	1.0uL		**Air Gap (s)**	1.0uL
2. Air Vol	1.0uL	1.0uL		**1. Air Vol Ndl**	1.1uL
2. Inject to	GC Inj 2	GC Inj 2		**Pre Cln Slv 1**	2
Pre Cln Slv 1	2	2		**Pre Cln Slv 2**	2
Pre Cln Slv 2	2	2		**Fill Speed**	5.0uL/s
Pre Cln Sp 1	0	0		**Pull Up Delay**	1.0s
Int Cln Slv 1	2	2		**Inject to**	GC Inj 1
Int Cln Slv 2	2	2		**Inject Speed**	5.0uL/s
Pst Cln Slv 1	2	2		**Pre Inj Del**	0ms
Pst Cln Slv 2	2	2		**Pst Inj Del**	0ms
Fill Volume	10uL	10uL		**Pst Cln Slv 1**	2
Fill Speed	2.5uL/s	2.5uL/s		**Pst Cln Slv 2**	2
Fill Stroke	0	0			
Pull Up Delay	500ms	500ms			
Inject Speed	10uL/s	10uL/s			
Pre Inj Del	0ms	0ms			
Pst Inj Del	0ms	0ms			

Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 1: A1016 at 0.500ug/mL Plot

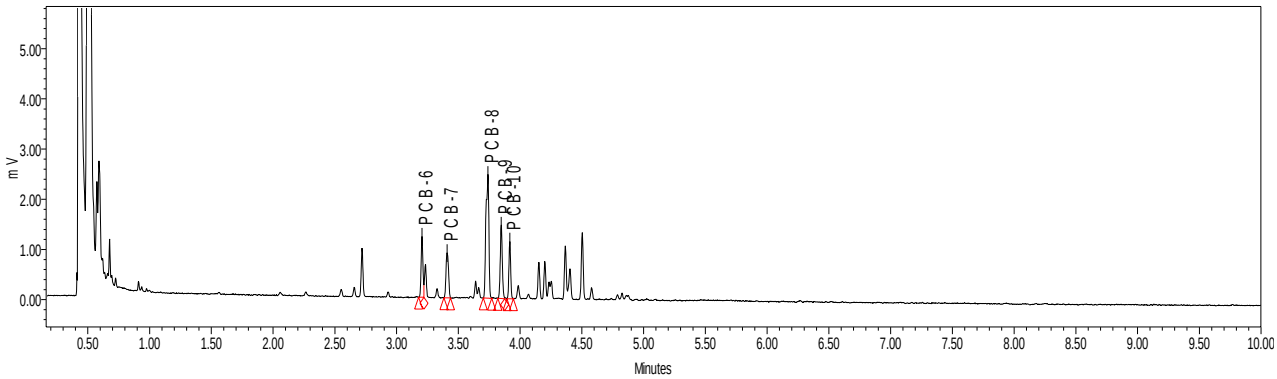
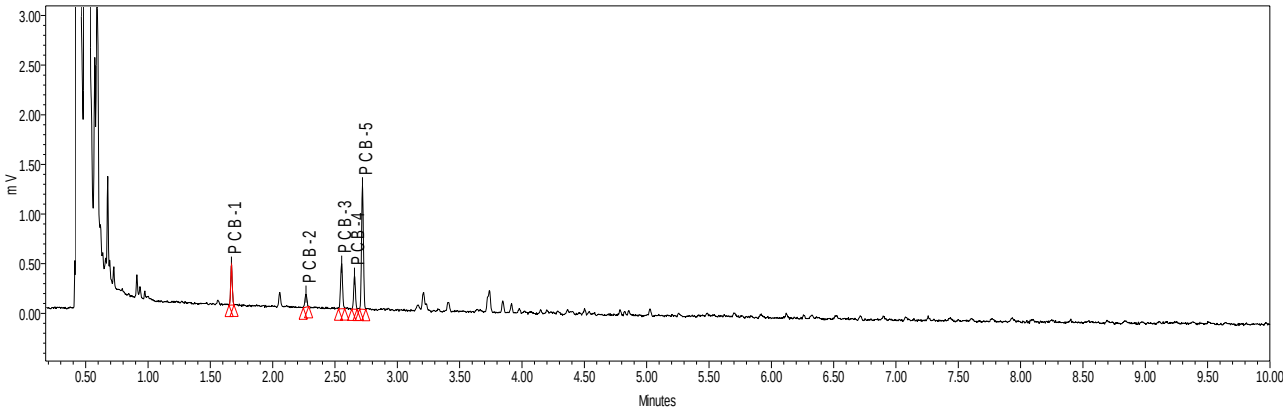


FIGURE 2: A1221 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 3: A1232 at 0.500ug/mL Plot

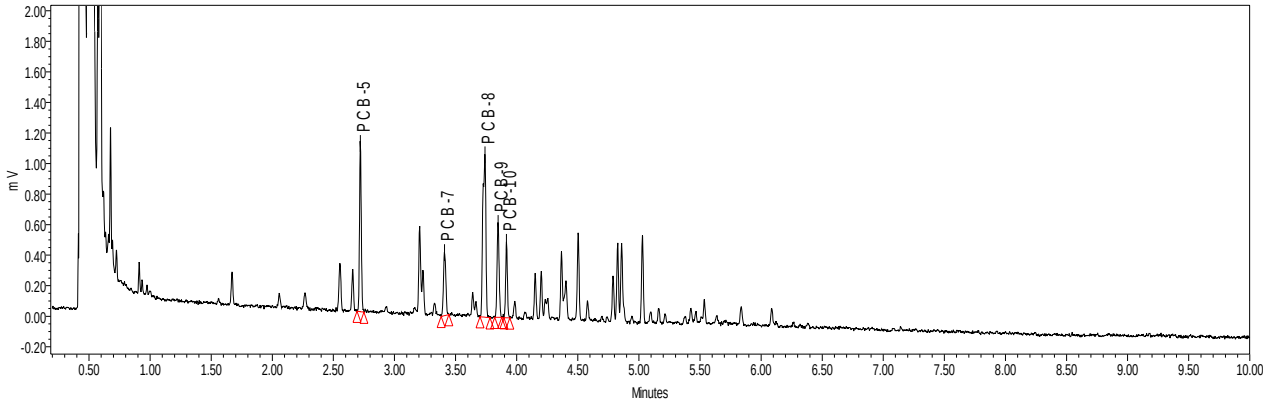
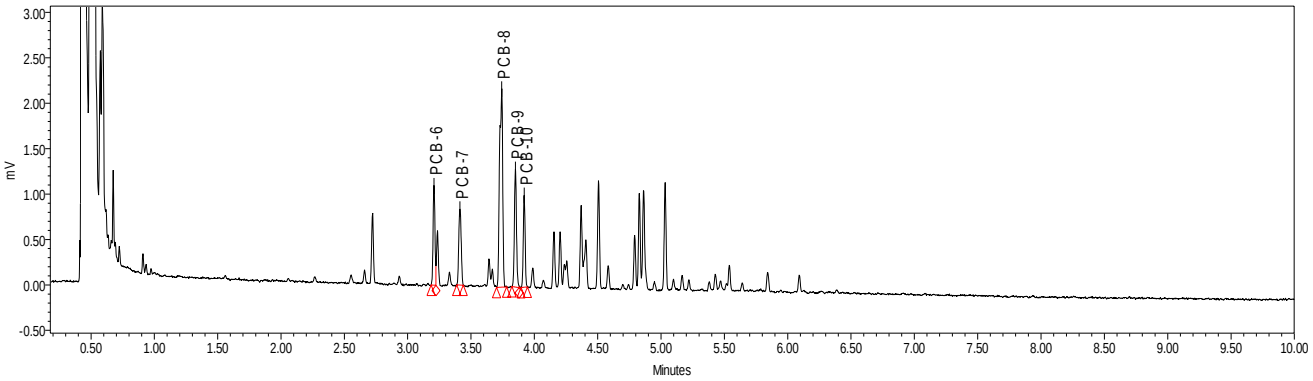


FIGURE 4: A1242 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 5: A1248 at 0.500ug/mL Plot

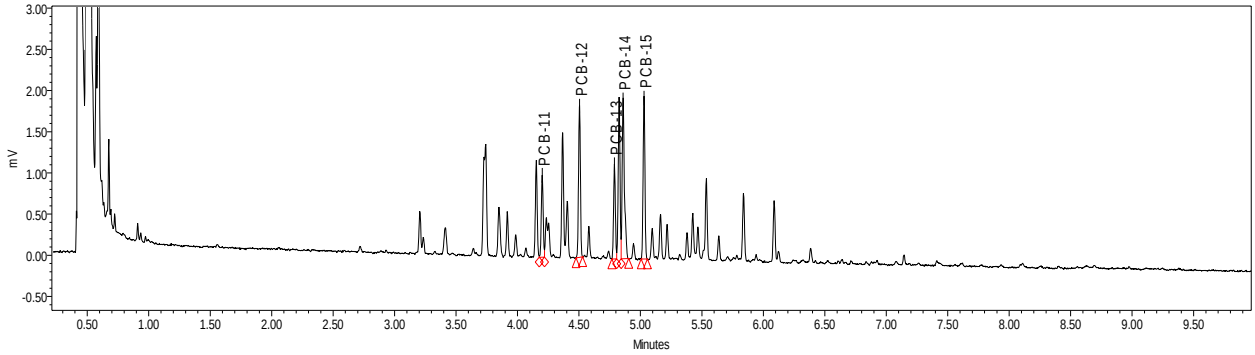
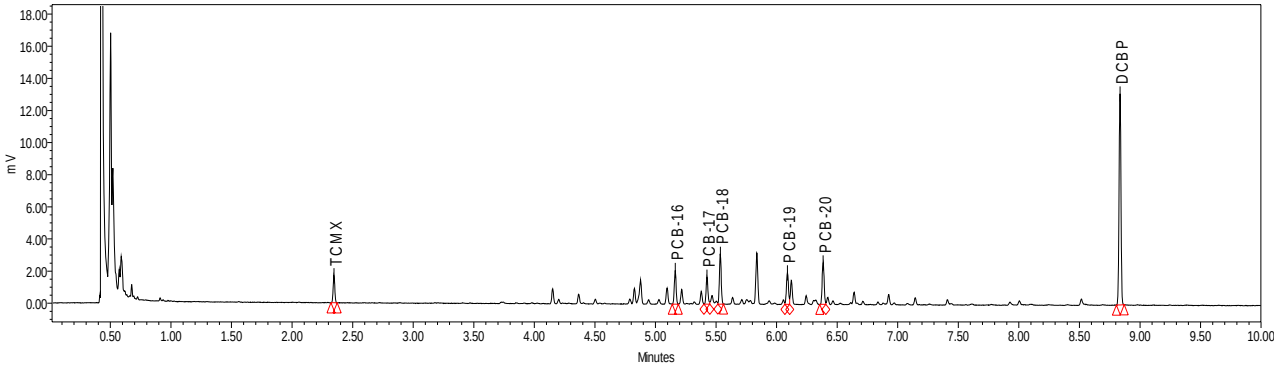


FIGURE 6: A1254 at 0.500ug/mL w/ TCMX & DCBP at 10/100ug/L Plot



Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 7: A1260 at 0.500ug/mL Plot

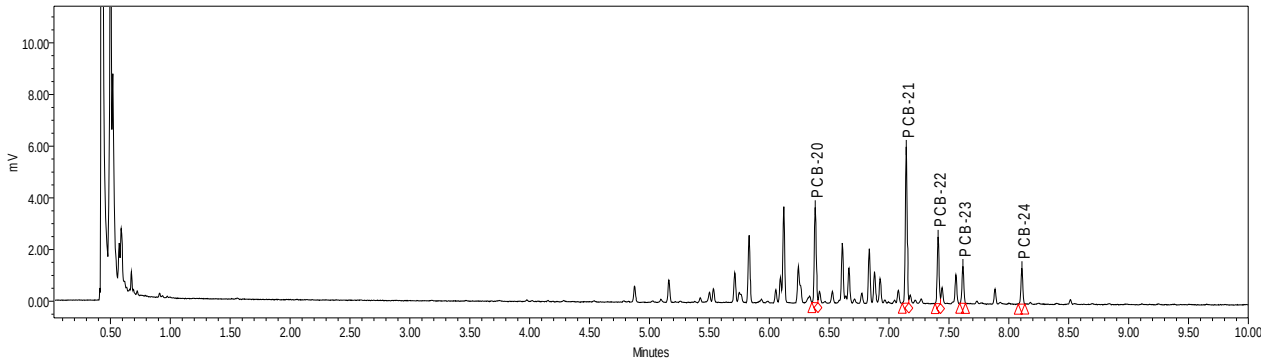
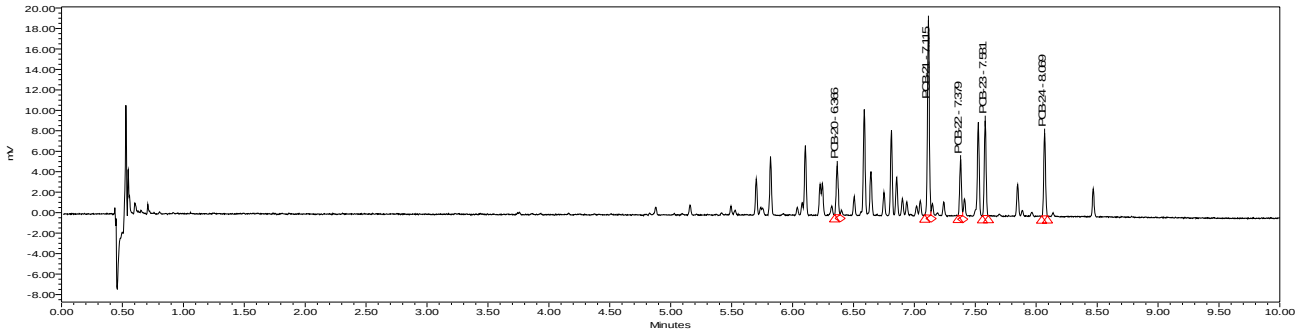
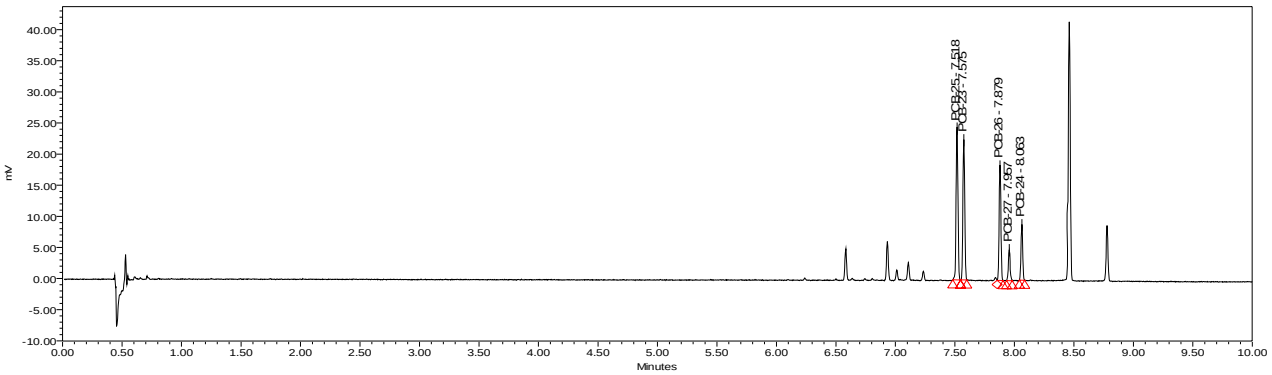


FIGURE 8: A1262 at 0.500ug/mL Plot



Attachment IV: Chromatograms (ZB-1MS Column/Hydrogen Carrier Gas) (continued)

FIGURE 9: A1268 at 0.500ug/mL Plot



P-20

Chain-of-Custody, Log-In, Tracking Procedures and Sample Containers (Rev. 4.9; 02/02/2016; GEN-003)

CHAIN-OF-CUSTODY, LOG-IN, TRACKING PROCEDURES AND SAMPLE CONTAINERS

1 PURPOSE

1.1 This SOP addresses the procedures for samples received by ECCS Mobile Lab Services, a division of Pace Analytical Services, Inc. fixed or onsite (ECCS), and the entry of the information into ECCS LIMS system, Prōmium Element® (LIMS).

2 SCOPE AND APPLICATION

2.1 This SOP is applicable to all samples received by ECCS. The SOP contains specifics for both fixed-based (Madison) and onsite laboratory operations.

3 ACCOUNTABILITY

3.1 The operations manager shall be accountable for this SOP. All employees of ECCS shall be responsible for the implementation of this SOP.

4 SAMPLE RECEIPT PROCEDURE

4.1 Samples arriving in the laboratory are typically accompanied by chain of custody (COC). The ECCS COC is shown in Figure 1.

4.1.1 Review the COC(s) accompanying the samples and see that adequate client identification, contact, and analytical request information has been entered.

4.1.2 If any of the information on the COC is in question, have the ECCS project manager contact the client to clarify.

4.1.3 Document any changes directly on the COC. Indicate who approved the changes, and make sure to date and initial.

4.1.4 Sign and enter the date and time received on the COC(s) in the spaces marked “Received by”.

4.1.5 Note if there were custody seals in the space provided.

4.1.6 Note the courier service used to ship the samples in the space provided. If the samples were picked up by an ECCS staff member write “pick-up”, and if the samples were dropped off by the client write “walk in”, “hand delivered” or “dropped off.”

4.1.7 Record the temperature of the temperature blank. For samples that require preservation at 4 °C, the acceptable range is “from just above freezing to 6 °C”. **If the temperature is outside of this range the ECCS Project manager/client must be notified. See Section 4.3.**

4.1.8 If samples are received without a temperature blank, record the temperature as effectively as possible from inside the cooler.

4.1.9 Samples that are delivered to the lab the same day as they are collected are likely not to have reached a fully chilled temperature. This is acceptable if there is evidence that chilling has begun and they are on ice.

4.1.10 Document the serial number and expiration date of the thermometer used to take the temperature on the COC.

4.2 Compare the labels on the samples received with the sample ID's on the COC. Any discrepancies should be noted in the comments section for the sample and immediately brought to the attention of the ECCS Project Manager/client. **In cases where the correct ID cannot be determined the sample ID on the COC shall be used. A comment should be added to the sample narrative field in LIMS that describes the problem and the resolution.**

4.3 Check the integrity of the samples. Any problems should be noted in the comments section and immediately brought to the attention of the ECCS Project Manager/client. Damaged containers, expired hold times, absent or incorrect preservation including receipt with no ice or temperatures over 6 °C, and air bubbles in VOC vials are examples of possible integrity issues. See Table 1 for sample containers, preservation and maximum holding times. **The client should be notified about any sample integrity issue. The client's response to a sample integrity issue must be documented on the COC or on a CAF and maintained in the work order folder. Sample integrity issues must also be documented in the sample narrative field in LIMS.**

4.3.1 **Verification of preserved sample pH at the time of receipt.** Preserved water samples must be checked via pH paper upon receipt to insure they were preserved to the correct pH. This is done using wide range pH paper. Shake the sample and dip a new, disposable, glass Pasteur pipette into the water. Touch the pipette tip, which will contain some water by capillary action to the pH paper. Compare the color of the strip to the guide on the pH box. **The pH should be <2 for acid preserved samples; >12 for samples preserved with NaOH; and pH 4-5 for n-methyl carbamate samples preserved with ChlorAC™.** Check the "Preservation Confirmed" box on the work order page in LIMS.

4.3.1.1 **Sample pH adjustment (if necessary).** Water samples with a lot of sediment may need additional preservation. In this event add small amounts (less than one mL) of the correct acid, either concentrated sulfuric acid or concentrated nitric acid, until the correct pH is obtained. The type of acid in the bottle should be indicated on the cap with a sticker. Sulfuric acid is used to preserve samples to be analyzed for nitrate+nitrite, ammonia, total kjeldahl nitrogen and total organic carbon. Nitric acid is used to preserve samples to be analyzed for metals. ChlorAC™ Buffer is used to preserve

samples to be analyzed for n-methyl carbamates by 8318. **If the sample pH is adjusted at the time of sample receipt, a comment to that affect should be added to the sample narrative field in LIMS and the client should be contacted.**

4.3.2 Water samples for VOCs should be checked to insure that no air-bubbles are present. Bubbles greater than 6 mm should be brought to the attention of the ECCS project manager/client. **If there are air bubbles greater than 6 mm in a VOC sample a corrective Action Form (CAF) must be prepared. The ECCS Project Manager and/or the client should be notified for a decision to proceed with the analysis. If bubbles >6 mm are present, the finding must be documented in the case narrative of the final report. The completed CAF should be included with the log-in paperwork that is routed to the analyst.**

NOTE: VOC water samples are preserved with 1:1 hydrochloric acid, but are not checked for pH during log-in. This would require opening the VOC vial, which would create head space in the vial and invalidate the sample.

4.4 The laboratory is considered a secured area with limited access. Any samples arriving outside of normal business hours are stored in either the unprocessed cooler or the annex refrigerator until they are logged in. Laboratory personnel are responsible for the care and custody of the samples from log-in until they are properly disposed of.

4.5 If the samples received are non-routine, contact the operations manager or project manager for guidance.

5 LOG-IN-PROCEDURE - (FOR FIXED OR ONSITE LABORATORY PROJECTS USING LIMS)

5.1 Log into LIMS.

5.2 Check to see if the client and project are in LIMS.

5.2.1 Click on Project Management in the task bar and then Projects.

5.2.2 Use drop down menu to select appropriate client. If the client does not exist see Section 5.3.

5.2.3 Look for appropriate project in the list of existing projects. If the project does not exist see section 5.4. If the project does exist see section 5.5.

5.3 Set up a new client in LIMS.

5.3.1 Click on Project Management in the task bar and then click on Clients.

5.3.2 Click on “add.”

5.3.3 Fill in the blanks in the client screen.

5.3.3.1 Most client types will be "Sampler by Project." Third parties needing an invoice are invoice only. Laboratories are either Network (the laboratory also uses Prōmium Element®) or Non-Network (the laboratory has a different LIMS system.)

5.3.3.2 The contact name needs to be put in the cells under Contacts. The address, phone numbers, etc. only need to be put in if they differ from the information at the top of the screen.

5.4 Set up the project in LIMS.

5.4.1 If the project for the samples being logged in does not exist in LIMS, it must be created by going to Project Management > Projects.

5.4.2 Click on either "add" or "copy" to begin creating the project. "Add" would be used to create a project from scratch and "copy" would be used if a similar project already exists that could be easily modified to fit the current project.

5.4.3 LIMS will prompt you to enter a name for the new project. Projects are usually named in the following manner: Name of the project – City, State (abbreviation). (Example Smith Ag Service – Madison, WI)

5.4.4 Fill in the blanks in the project screen. Information should be found on the COC.

5.4.5 Custom compound lists can be created by right clicking on the analysis name and clicking "Edit Custom Analysis." When creating a custom compound list be sure to edit all matrices. A note should also be made in the comments section as to how the list was customized. Besides analytes, many other fields are editable for specific projects including reporting limits, units, control limits, CLP qualifiers, wet weight vs. dry weight basis, etc.

5.5 Create Work Order.

5.5.1 Click on Sample Control in the task bar and then click on Work Order.

5.5.2 Click "add" and complete all of the blanks in the work order screen, then verify for accuracy. This is a crucial step in the process to properly document sample receipt conditions, the lab project manager responsible for these samples, as well as the actual turnaround time promised to the client. Here are some guidelines:

- Don't include additional analytes or analyses in the comments section of the work order or "sample comments". They should be included in the "Analysis" section as a custom analysis if needed.

- List all the analyses from the COC in the Analysis box.

- Record the condition of samples. Specific problems like broken sample containers must be documented in the case narrative section by clicking on the icon labeled "CSN."

- Assign a specific lab project manager who is accountable for the samples while in the lab and in contact with the client regarding the samples' analysis status.

5.5.3 Click "Save" to save changes, and then "Samples." This will take you to the samples screen. The same screen can also be accessed under Sample Control in the task bar and then Samples.

5.6 Enter Sample Data

5.6.1 Click "Add" to begin entering sample data.

5.6.2 Fill in the blanks in the sample data screen. Check all of the tabs for accuracy and completeness in both the center of the screen ("Sample Information" and "Containers") and on the right hand side of the screen ("Sample", "Details", and "Location.")

5.6.3 Click "Save" to save changes.

5.6.4 Additional samples can be added using either the "Add" or "Copy" buttons.

5.7 Subcontracted Samples

5.7.1 Subcontracting samples to another lab

5.7.1.1 If ECCS does not do a test required by the client, the sample(s) must be subcontracted.

5.7.1.2 If NELAC or state specific certification is required, the project manager is responsible for ensuring that the subcontracted laboratory has the correct certifications.

5.7.1.3 Analyses can be designated as subcontracted in the project or subcontracted at the time of log-in. To subcontract at the time of log-in, right click on the appropriate analysis in the lower half of the screen and click on "Subcontract." This will prompt you to pick the subcontract lab.

5.7.1.4 The location of the sample containers to be subcontracted should also be changed to reflect the correct subcontract lab.

5.7.2 Receiving sub-contracted sample information from another Element® lab

5.7.2.1 On the work order page in LIMS click on "Import."

5.7.2.2 Select the transfer file from the "Transfer In" folder. Once a file is selected, it will no longer be available to import.

5.7.2.3 Match the Remote Client and Project with the Local Client and Project.

5.7.2.4 Also match the Analyses, Matrices, and Containers.

5.7.2.5 Finally select done and log-in the samples as described in Sections 5.6 thru 5.10. Sample Names, etc. will be imported with the transfer file minimizing the amount of information that needs to be entered.

5.8 Print Work Order, Sample Labels, and Subcontract Order.

5.8.1 After all samples have been entered, click the button with the printer icon.

5.8.2 The first prompt will be for printing the work order. The print format should be wko_default.rpt.

5.8.3 Click on the close button in the upper right of the dialog box. This will move you to the Sample Labels print dialog.

5.8.4 The print format for labels should be lsx_m@5160.rpt. If all labels do not need to be printed you can highlight just those needed. To generate labels for lids, use template lsx_m@eccs.rpt.

5.8.5 Click on the close button in the upper right of the dialog box. If any samples were subcontracted it will prompt you to print the Subcontract Order. If no samples are being subcontracted the dialog box will close. Samples being sub-contracted to an in-network lab may require a transfer out file to be generated. This is done by clicking on "Export" from the work order page. Because of anti-virus software, the .mdb file that is generated will need to be zipped prior to e-mailing this to the in-network laboratory.

5.8.6 Review your output to verify that all sample descriptions, dates, analysis, etc. were entered correctly.

5.8.7 Affix the sample labels to the appropriate jars and lids (for soil jars).

5.8.8 For samples received at the fixed base laboratory, the original work order and COC are placed in a manila folder labeled with the work order, client, and project (if applicable), and the number of samples under the work order on the folder tab. This becomes the final report folder to include all project-related correspondence.

5.8.9 **Route the folder containing the COCs and the printed work order to the Operations Manager, Lab Project Manager, or a designated person for review of the LIMS entry.** The reviewer should double-check that the proper LIMS analysis is assigned to the samples. Many analytes can be analyzed by

different analysis codes (e.g. 8151 GC/MS vs. HPLC, 8260 by P&T vs. DI, or Full Scan vs. SIM). Documentation on the COC and any CAFs should also be reviewed for clarity and to verify that any issues have been properly recorded and resolved. Following this review the reviewer should update the LIMS status to "Available."

5.8.10 Scan the COC and electronically file it in the appropriate section of the Fixed Laboratory Section of the P drive.

5.9 For samples received at the fixed base laboratory, the COC and work order needs to be copied and stapled together for each analysis. Highlight the applicable analysis for each copy as each analysis needs a separate copy. These copies are placed in the clear bin labeled "Needs to be Extracted" on the wall in the extraction area.

5.10 For onsite laboratory projects not using LIMS, or if LIMS is not available, assign a sample number based on the ECCS project number and sequential numbering, for example 2389-1,2,3, etc. Differentiate the containers for each sample using the alpha characters a, b, c, d, etc. For some projects differentiating the containers of each sample may not be necessary, as long as the practice is not required at the site by accrediting bodies, regulatory authorities, or the client.

6 SAMPLE STORAGE

6.1 Samples are put in the correct refrigerator as soon as possible to reduce their time at ambient temperature. In the Madison laboratory most samples are stored in the unprocessed refrigerator with the exception of VOC samples, which are stored in the Annex refrigerator.

6.2 Samples to be subcontracted and the subcontract order are packed in a cooler with ice and sent to the appropriate laboratory. Samples must be delivered to the subcontract laboratory the following day to ensure that they are received on ice. Therefore, samples received late on Thursdays or on Fridays may be held and shipped on Monday if the remaining holding time allows for this delay.

7 SAMPLE MANAGEMENT

7.1 All samples are managed for disposal in accordance with Federal, State and local regulations.

7.2 In the fixed-based laboratory sample management information is documented in an excel file called 0Disposal.xxxx where xxxx is the current year. (example 0Disposal.2007)

7.2.1 Water samples are disposed no sooner than one month from receipt.

7.2.1.1 Water samples are disposed by dumping them down the drain through a carbon filter. The cold water faucet is left running at a medium flow to

flush the drain during the disposal and for at least 10 minutes following disposal.

7.2.2 Soil samples are disposed no sooner than six months from date of receipt.

7.2.3 After keeping soil samples in cold storage for at least a month they can be moved to warm storage. The soil samples are organized into labeled boxes and stored off site.

7.2.3.1 Before being organized and boxed, soil samples are queried in LIMS and any samples with detections above the appropriate hazard level are segregated.

7.2.3.1.1 This is done in Sample Control/Disposal

7.2.3.1.2 Samples above the appropriate hazard level have information in the "Hazard Info" column.

7.2.4 Soil samples are composited and tested before disposal. The sample to be tested is obtained by taking a subsample from five random jars in each box to be disposed. The subsamples are then composited and tested for 8141, 8151 and 8081 pesticides and/or specific landfill testing requirements.

7.2.5 Any soil samples submitted to the lab that are hazardous as segregated in Section 7.2.3 or as tested in Section 7.2.4, are kept isolated and will be disposed as hazardous waste or returned to the client.

7.3 For onsite laboratory operations the samples are returned to the client at the conclusion of the project prior to returning to the Madison laboratory. In rare cases, when the samples cannot be returned to the client, the samples are returned to the Madison laboratory for sample management according to Section 7.2.2.

8 SAMPLE CONTAINERS

8.1 Sample Container Cleanliness

8.1.1 Sample containers are purchased pre-cleaned.

8.1.2 Sample container cleanliness is checked during limit of detection studies. See SOP "GEN-019 Limit of Detection (LOD) Determinations".

8.1.3 Client test results are monitored continuously for anomalies that could be attributed to sample container cleanliness. Any anomaly is immediately investigated and documented in the "QA Tracking" Excel file.

8.2 Sample Container Preparation

8.2.1 When sending sample containers to a client, make sure a temperature blank is included in the cooler.

8.2.2 Commonly Used Sample Containers

8.2.2.1 4 ounce, wide mouth amber jar with a Teflon lined lid. (ESS part number 0125-0055-PC or equivalent)

8.2.2.2 Plastic specimen cups with lids. (VWR part number 89107-308 (cups) 89107-312 (lids) or equivalent)

8.2.2.3 125 mL plastic bottles. (VWR part number 10625-9005 or equivalent)

8.2.2.4 1 liter, narrow mouth amber bottle with a Teflon® lined lid. (ESS part number 1000-0150-PC or equivalent)

8.2.2.5 Lock 'n' Load ™ syringe. (ESS part number LL5035SRNG or equivalent)

8.2.2.6 40 mL unpreserved VOC vials. (LSDC part number 23150-40D or equivalent)

8.2.2.7 40 mL VOC vials preserved with 1:1 HCl. (ESS part number 4050-0300-PC or equivalent)

8.2.3 Upon client request, ECCS provides appropriate sampling containers for all analysis listed in Table 1. Containers for tests that are to be subcontracted should be obtained from the subcontract lab.

8.2.4 List of appropriate containers

8.2.4.1 Nitrate+nitrite, ammonia, total kjeldahl nitrogen, total phosphorus

8.2.4.1.1 Soil samples are usually collected in 4 oz amber glass jars. It is also appropriate for samples to be collected using plastic specimen cups or plastic bags.

8.2.4.1.2 Water samples are collected in 125 mL plastic bottles that contain approximately 0.1 mL of concentrated sulfuric acid or in 250 mL plastic sulfuric acid preserved bottles obtained from the subcontract laboratory..

8.2.4.1.3 Preservative for water samples is added using a repipetter. Repipet 0.1 mL of concentrated sulfuric acid into an empty 125 mL plastic bottle. Cap the bottle tightly. Affix a sticker indicating that the bottle has been pre-preserved with sulfuric acid.

8.2.4.2 Total organic Carbon

8.2.4.2.1 Soil samples are usually collected in 4 oz amber glass jars. Alternate size jars can be substituted, but they must be glass and amber colored.

8.2.4.2.2 Water samples are collected in 125 mL amber glass containers pre-preserved with sulfuric acid. These are obtained from the subcontract laboratory.

8.2.4.3 Chlorinated pesticides and PCB's, nitrogen phosphorus pesticides, chlorinated acid herbicides, urea carbamate herbicides

8.2.4.3.1 Soil samples are usually collected in 4 oz amber glass jars. Alternate size jars can be substituted, but they must be glass and amber colored.

8.2.4.3.2 Water samples are collected in 1 liter amber bottles. For each set of bottles ordered two extra amber liters are provided to be used for matrix spiking. The client is asked to fill the two additional amber liters at one of their sample points.

8.2.4.4 N-methyl carbamate pesticides

8.2.4.4.1 Soil samples are usually collected in 4 oz amber glass jars. Alternate size jars can be substituted, but they must be glass and amber colored.

8.2.4.4.2 Water samples are collected in 1 liter amber bottles that contain 10-15 mL of ChlorAC™ buffer.

8.2.4.4.3 Preservative for water samples is added using a graduated cylinder. Measure between 10 and 15 mL of ChlorAC™ buffer and add to an empty 1 L amber bottle. Indicate on the label that the bottle has been pre-preserved with ChlorAC™.

8.2.4.5 Volatile organic compounds

8.2.4.5.1 Soil samples are collected using either the Lock 'n' Load ™ sampling device or by weighing the samples in the field. Both methods provide approximately 10 grams of sample. The soil sample is then transferred by the client into a pre-tared VOC vial containing 10 mL of methanol.

8.2.4.5.2 To prepare the pre-tared vials add 10 mL of methanol to an empty vial using a Repipetter. Then affix a sample label and a methanol label. Weigh the vial, with cap and labels and record the weight on the label. See Form GEN-003-1

8.2.4.5.3 Water samples are collected in two or three 40 mL VOC vials that are purchased pre-preserved with 1:1 HCL.

8.2.4.6 Chloride

8.2.4.6.1 Soil samples are usually collected in 4 oz amber glass jars. It is also appropriate for samples to be collected using plastic specimen cups.

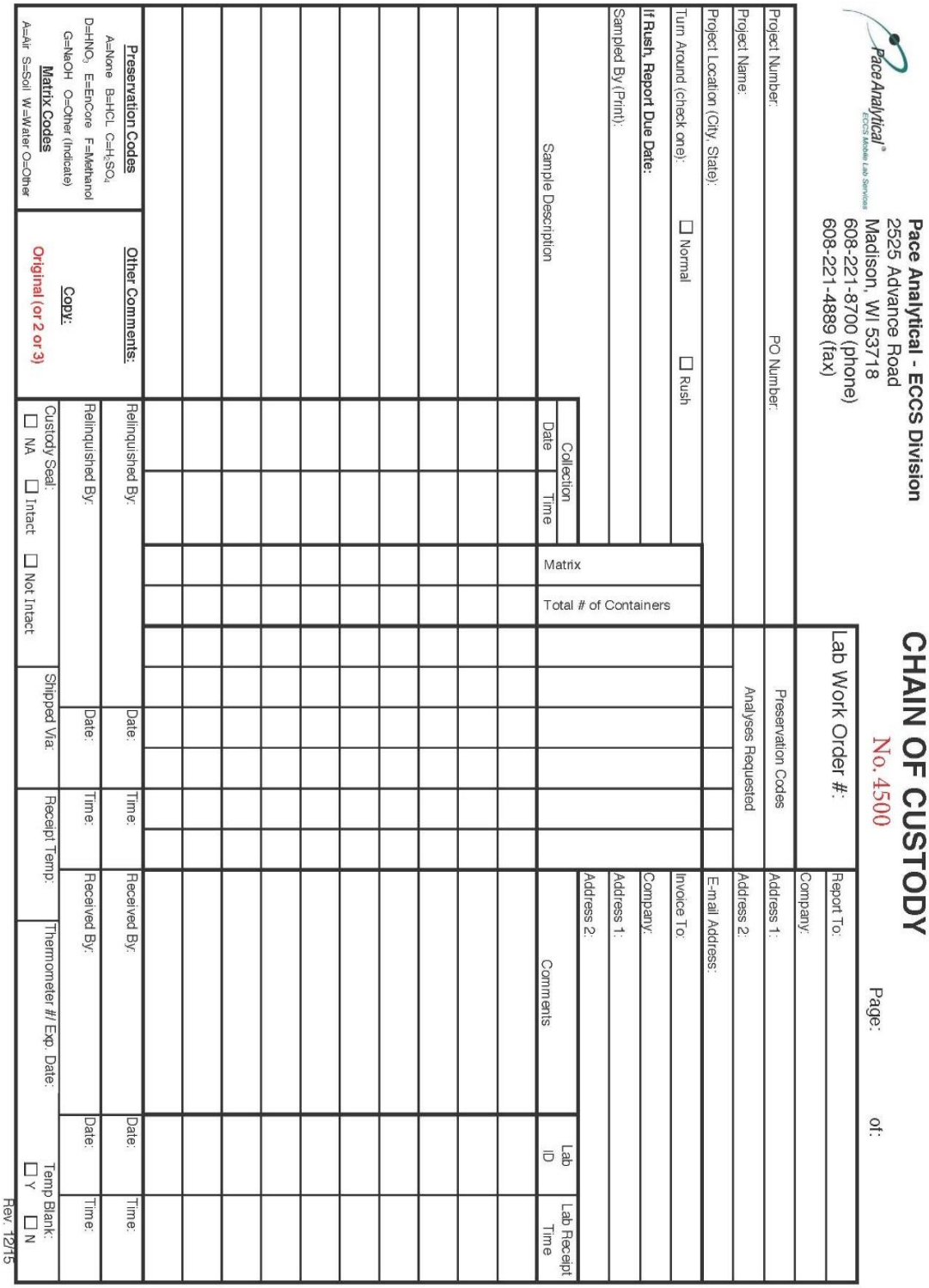
8.2.4.6.2 Water samples are collected in 125 mL unpreserved plastic bottles.

8.2.4.7 Metals

8.2.4.7.1 Soil samples are usually collected in 4 oz amber glass jars. It is also appropriate for samples to be collected using plastic specimen cups.

8.2.4.7.2 Water samples are collected in 250 mL plastic bottles that are pre-preserved with nitric acid. These are obtained from the subcontract laboratory.

FIGURE 1



Pace Analytical - ECCS Division
2525 Advance Road
Madison, WI 53718
608-221-8700 (phone)
608-221-4889 (fax)

CHAIN OF CUSTODY

No. 4500

Page: of:

Lab Work Order #:

Project Number: PO Number:
Project Name:
Project Location (City, State):
Turn Around (check one): ☐ Normal ☐ Rush
If Rush, Report Due Date:
Sampled By (Print):

Report To:
Company:
Address 1:
Address 2:
E-mail Address:
Invoice To:
Company:
Address 1:
Address 2:

Preservation Codes
Analyses Requested

Sample Description	Collection Date	Collection Time	Matrix	Total # of Containers				Comments	Lab ID	Lab Receipt Time

Preservation Codes
A=None B=HCL C=H_2SO_4
D=HNO_3 E=EnCore F=Methanol
G=NaOH O=Other (Indicate)

Matrix Codes
A=Air S=Soil W=Water O=Other

Other Comments:

Copy:
Original (or 2 or 3)

Relinquished By: Date: Time: Received By: Date: Time:
Relinquished By: Date: Time: Received By: Date: Time:
Custody Seal: ☐ NA ☐ Intact ☐ Not Intact
Shipped Via: Receipt Temp: Thermometer #/ Exp. Date:
Temp Blank: ☐ Y ☐ N

Rev. 12/15

TABLE 1
Sample Containers, Test Method, Preservation, and Holding Times

SOIL SAMPLES	Method	Container	Chemical Preservation	Holding Time
Petroleum Hydrocarbons (DRO)	WI DRO, 8015	4 oz. a, g	none	14 days
Petroleum Hydrocarbons (GRO)	WI GRO, 8015	40 mL g	1:1 methanol[1]	14 days
Volatile Organics	8260	40 mL g	1:1 methanol[1]	14 days
Volatile Organics	8260	Encores	none	48 hours
Semi-volatile Extractable Organics	8270	4 oz. a, g	none	14 days
Organochlorine Pesticides	8081	4 oz. a, g	none	14 days
Nitrogen/phosphorus Pesticides	8141, 8270	4 oz. a, g	none	14 days
Urea Carbamates	8321	4 oz. a, g	none	14 days
n-Methyl Carbamates	8318	4 oz. a, g	none	14 days
Acid Herbicides	8151	4 oz. a, g	none	14 days
PCBs	8082	4 oz. a, g	none	14 days
PAHs	8270	4 oz. a, g	none	14 days
Explosives	ECCS	4 oz. a, g	none	14 days
Metals	6010	4 oz. a, g	none	6 months
Mercury	7471	4 oz. a, g	none	28 days
% Solids	ECCS	Spec. cup, p	none	14 days
pH	EPA 9045	Spec. cup, p	none	NA
Nitrate or Nitrite	EPA 353.2	Spec. cup, p	none	28 days
Nitrate+Nitrite	EPA 353.2	Spec. cup, p	none	28 days
Ammonia	EPA 350.1	Spec. cup, p	none	28 days
WATER SAMPLES				
Petroleum Hydrocarbons (DRO)	WI DRO, 8015	1 L a, g	none	7 days
Petroleum Hydrocarbons (GRO)	WI GRO, 8015	2-40 mL[2], g	HCl to pH<2	14 days
Volatile Organics	8260	2-40 mL[2], g	HCl to pH<2	14 days
Semi-volatile Extractable Organics	8270	none	none	7 days
Organochlorine Pesticides	8081	none	none	7 days
Nitrogen/phosphorus Pesticides	8141, 8270	none	none	7 days
Urea Carbamates	8321	none	none	7 days
n-Methyl Carbamates	8318	none	10ml ChlorAC	7 days
Acid Herbicides	8151	none	none	7 days
PCBs	8082	none	none	7 days
PAHs	8270	none	none	7 days
Explosives	ECCS	none	none	7 days
Metals	6010	none	HNO_3 to pH<2	6 months
Mercury	7471	none	HNO_3 to pH<2	28 days
pH	EPA 9045	none	none	NA
Nitrate or Nitrite	EPA 353.2	none	none	48 hours
Nitrate+Nitrite	EPA 353.2	none	H_2SO_4 to pH<2	28 days
Ammonia	EPA 350.1	none	H_2SO_4 to pH<2	28 days

All samples should be cooled to 4°C during shipment to the lab and while in storage at the lab.

1 40ml VOC vials with Teflon® septum. Add the sample to methanol at a 1:1, g:mL ratio.
2 40ml VOC vials with Teflon® septum.

a= amber; g=glass: p=plastic NA= Should be analyzed immediately (within 15 minutes).

Note: This guide is intended as a guide only. Refer to SW-846 or EPA methods for official method requirements.

FORM GEN-003-1

Log Page Number_____

VOC SOIL WEIGHING LOG

Preparation Date:_____ **Received Date:**_____

Prepared By:_____ **Repipettor Checked:** **Received By:**_____

Balance ID:_____ **Yes**_____ **Balance ID:**_____

ECCS Sample Number	Sample ID	Initial Jar Wt. (g)	Final Jar Wt. (g)	Initial MEOH (ml)	Wt. Soil (g)	Final MEOH (ml)

	Manufacturer	Lot Number	Density @ 20°C
Methanol, P&T Grade			0.791 g/ml

Comments:_____

The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

Reviewed By:

01/26/16

Karl Olm, Operations Manager — Date

01/26/16

Patrick Letterer, Quality Manager — Date

Approved By:

01/26/16

Nick Nigro, General Manager — Date

P-21

Waste Handling and Management
(Rev. 01; 03/31/2017; MAD-W-002)

Pace Analytical

STANDARD OPERATING PROCEDURE

WASTE HANDLING AND MANAGEMENT

Reference Methods: N/A

Local SOP Number:	SOP-MAD-W-002-rev.01
Effective Date:	Date of Final Signature
Supersedes:	ECCS GEN-003, GEN-026, GEN-027
SOP Template Number:	SOT-ALL-W-002-rev.06

APPROVALS

Laboratory General Manager	3/31/17 Date
Laboratory Quality Manager	03/31/17 Date
Laboratory Hazardous Waste Coordinator	3/31/17 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose/Identification of Method

1.1. Pace Analytical Services, LLC (Pace) acknowledges its obligation to the responsible management of the environment and its resources. Pace Management is committed to operating in such a way that meets or exceeds the state and federal laws governing waste management and encourages the use of best practices to reduce, reuse and recycle waste material where possible. This Standard Operating Procedure (SOP) documents the systems, processes and procedures that Pace Analytical Services, LLC – Mobile Laboratory Services located in Madison, WI (Pace – Madison) uses to manage generated wastes.

1.2. It is Pace's policy to minimize the amount of hazardous waste it produces and to reduce the hazardous properties of those wastes whenever practical within regulatory compliance. This can be achieved by periodic auditing of all processes producing hazardous waste; reduction of sample volume delivered by the client; return of excess sample material to clients whenever practical and economical; investigation of new technologies that might require smaller volumes of sample, or produce fewer or less hazardous by-products; implementation of lab cleaning procedures that reduce the volume of cleaning residue; recycling of hazardous materials; and investigation of new treatment technologies that are comprehensively destructive or are effective in reducing the volume or hazardous qualities of the wastes produced.

2. Summary of Method

2.1. Pace facilities that generate waste must initially contact the EPA to obtain an ID number. Each unique type of generated waste is classified and characterized into waste streams according to procedures in 40 CFR 261. The amount of waste the facility generates determines the Generator Status of a lab, which in turn determines how long and how much waste can accumulate. Pace – Madison is ultimately responsible for the waste it generates, and is required to obey any and all regulations during the process of creating, accumulating, disposing, and releasing waste to a TSDF for final disposal. Documentation is kept to prove all regulations have been obeyed.

3. Scope and Application

3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel responsible for all aspects of waste handling and management.

3.2. This SOP is applicable to all processes at Pace – Madison that involve generated waste, and is designed to assist its operations in adhering to regulations set forth in the following federal statutes: Resource Conservation and Recovery Act (RCRA), Clean Water Act (CWA), Toxic Substances Control Act (TSCA), and DOT Title 49, and Transportation (parts 100-199). Particular attention is given to local pretreatment standards covering discharges to publicly owned treatment works (POTW) when performing elementary neutralization on acidic and basic waste. The local standards are based in part upon provisions in the National Pretreatment Standards and Prohibited Discharge Standards.

3.3. The degree to which RCRA regulations apply to Pace facilities is dependent upon the generator status of the operation. Under the federal rules (state requirements may be more stringent or give the classes a slightly different name) there are three different classes of hazardous waste generators based upon the amount of waste generated in a month to month time frame.

3.4. **Waste Generator Class Limits:**

Hazardous Waste Generator Class	Quantity of Hazardous Waste Generated per Month	Generated Monthly Acute Hazardous Waste	Maximum Allowable Hazardous Waste Quantity on-site	Maximum Permitted Waste Accumulation Time
Cond. Exempt Small Quantity	<100kg	<1 kg	<1000kg	Unlimited
Small Quantity	100-1000kg	<1 kg	<6000kg	180 days (270 days if the waste must be sent >200 miles to TSDF)
Large Quantity	>1000kg	>1kg	Unlimited	90 days

3.5. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. Not applicable to this SOP.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Not applicable to this SOP.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. **Acutely Hazardous Waste** - A waste which is hazardous as identified with an (H) Hazard Code in the lists of Hazardous Waste in 40 CFR Part 261, Subpart D, Sections 261.30, 261.31 and 261.33.

8.3. **Animal and Plant Health Inspection Service (APHIS)** - an agency of the USDA responsible for protecting animal health, animal welfare, and plant health. APHIS is the lead agency for collaboration with other agencies to protect U.S. agriculture from invasive pests and diseases.

8.4. **Clean Air Act** - The Federal Clean Air Act, 42 U.S.C. 7401, and amendments thereto amending 42 U.S.C. 1857 et.seq.

8.5. **Conditionally Exempt Small Quantity Generator (CESQG)** - A generator who produces no more than 100 kilograms of hazardous waste or one kilogram of acutely hazardous waste (or a total of 100 kilograms of any residue or contaminated soil, waste or other debris resulting from the cleanup of a spill, into or on any land or water, or any acute hazardous waste) in a calendar month. The total amount of hazardous waste which may be accumulated on-site is 1000 kilograms. Wisconsin recognizes conditionally exempt small quantity generator (CESQG) status as very small quantity generator status (VSQG).

8.6. **Confined Space** – A space that is large enough and so configured that an employee can bodily enter and perform assigned work; and has limited or restricted means for entry or exit (for example, tanks, vessels, silos, storage bins, hoppers, vaults, and pits are spaces that may have limited means of entry); and is not designed for continuous employee occupancy.

8.7. **Container** - Any device material is stored, transported, treated, disposed of, or otherwise handled.

8.8. **Contingency Plan** - A document setting out an organized, planned, and coordinated course of action to be followed in case of fire, explosion, or release of hazardous waste or hazardous waste constituents which could threaten human health or the environment.

8.9. **Designated Hazardous Waste Storage Area** - Area used to hold hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere. This is the storage area into which hazardous waste from the laboratory (e.g., satellite waste) is moved.

8.10. **DOT** - The United States Department of Transportation.

8.11. **DTSC** – Department of Toxic Substances Control.

8.12. **Elementary Neutralization Unit** – A device which: (1) is used for neutralizing wastes which are hazardous only because they exhibit the corrosivity characteristic defined in 40 CFR 261.22 or are listed in Subpart D of Part 261; and (2) meets the definition of tank, container, transport vehicle, or vessel in 40 CFR 260.10.

8.13. **EPA** - The United States Environmental Protection Agency.

8.14. **EPA Hazardous Waste Number** - The EPA number assigned to each EPA hazardous waste identified in 40 CFR Part 260, Subpart D - Lists of Hazardous Wastes.

8.15. **EPA Identification Number** - The site-specific number assigned to each generator, transporter, and TSDF upon approval of a notification form.

8.16. **Federal Clean Water Act** - 33 U.S.C. 1251, et. Seq.

8.17. **Foreseeable Emergency** - Any fire, explosion, or sudden or non-sudden release of hazardous waste or hazardous waste constituents to the air, soil, or surface water, which could threaten human health or the environment.

8.18. **Generator** - Any person, by site who owns or operates a facility where hazardous waste is generated, i.e. Pace Analytical Services (Pace).

8.19. **Hazardous Waste Coordinator** - The Pace employee responsible for creating, guiding, and implementing all hazardous waste management operations.

8.20. **Hazardous Waste** - As defined in 40 CFR Part 261, Subparts B and C, a solid, semi-solid, liquid or contained gaseous waste, or any combination of these wastes.

8.20.1. Which, because of either quantity, concentration, physical, chemical, or infectious characteristics may:

8.20.2. Cause or contribute to an increase in mortality or an increase in irreversible or incapacitating reversible illness; or

8.20.3. Pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, disposed of or otherwise mismanaged.

8.20.4. Or which has been identified as having a characteristic of hazardous waste by the EPA using the criteria established under 40 CFR Part 261, Subpart C, or as listed under Sections 261.31, 261.32, 261.33, and 261.34. Such wastes include, but are not limited to, those which are reactive, toxic, corrosive, ignitable, irritants, strong sensitizers or which generate pressure through decomposition, heat or other means. Such wastes do not include radioactive substances that are regulated by the Atomic Energy Act of 1954, as amended. A waste is considered hazardous if it is listed or it fits into one of four categories. These categories are as follows:

8.20.4.1. Ignitable – (40 CFR 261.21, Waste Code D001) A flash point of less than 60°C/140°F.

8.20.4.2. Corrosive – (40 CFR 261.22, Waste Code D002) A pH of less than 2.0 or greater than 12.5.

8.20.4.3. Reactive – (40 CFR 261.23, Waste Code D003) Reactive wastes exhibit one or more of the following characteristics:

8.20.4.3.1. It is unstable and can undergo a violent change without detonating.

8.20.4.3.2. It can react violently with water.

8.20.4.3.3. When mixed with water it can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

8.20.4.3.4. It is cyanide or sulfide bearing waste that, when exposed to pH conditions between 2.0 and 12.5, can generate gases, vapors, or fumes that can present a danger to human health or the environment.

8.20.4.3.5. It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.

8.20.4.3.6. It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.

8.20.4.3.7. It is a forbidden explosive as defined in 49 CFR 173.51, or a Class A explosive as defined in 49 CFR 173.53, or a Class B explosive as defined in 49 CFR 173.88.

8.20.4.4. Toxic – (40 CFR 261.24, Waste Codes D004-D043) A solid waste that contains a toxic concentration of a contaminant listed in 40 CFR 261.24, Table 1. A toxic waste is given any and all D-codes that apply to the particular material.

8.21. **Hazardous Waste Constituent** - A substance, compound, or element listed as hazardous waste in EPA 40 CFR 261.

8.22. **Lab Pack Material** - A hazardous waste that does not match a listed Pace waste stream category.

8.23. **Large Quantity Generator (LQG)** - Any generator who generates at a rate greater than 1000 kilograms of hazardous waste per month.

8.24. **Manifest** - As defined in 40 CFR Part 262, Subpart B, namely "the form used for identifying the origin, quantity composition, routing and destination of hazardous waste".

8.25. **Plant Protection and Quarantine (PPQ)** – A program within APHIS which attempts to safeguard agriculture and natural resources in the U.S. against the entry, establishment, and spread of animal and plant pests and noxious weeds.

8.26. **Regulated Soil** – Soil from foreign countries, U.S. territories and areas within states that are under Federal quarantine that can be moved into or through continental U.S. only if conditions and safeguards prescribed by the USDA and APHIS are met.

8.27. **Sample** - Except as provided below in 8.27.7.2.3, any solid waste, water, soil, or air that is collected for the sole purpose of being tested to determine its characteristics or composition.

8.27.1. Samples are not subject to any requirements of 40 CFR Part 261.5 or Parts 262 through 267 or Part 270 or Part 124 or to the notification requirements of Section 3010 of RCRA, when:

8.27.1.1 The sample is being transported to a laboratory for the purpose of testing; or

8.27.1.2 The sample is being transported back to the sample collector after testing; or

8.27.1.3 The sample is being stored by the sample collector before transport to a laboratory for testing; or

8.27.1.4 The sample is being stored in a laboratory before testing; or

8.27.1.5 The sample is being stored in a laboratory after testing but before it is returned to the sample collector; or

8.27.1.6 The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action where further testing of the sample may be necessary).

8.27.2. In order to qualify for the exemption in 8.27.1.1 and 8.27.1.2 above, a sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must:

8.27.2.1. Comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements; or

8.27.2.2. Comply with the following requirements if the sample collector determines that DOT, USPS, or other shipping requirements do not apply to the shipment of the sample:

8.27.2.2.1. Assure that the following information accompanies the sample:

8.27.2.2.1.1. The sample collector's name, mailing address, and phone number;

8.27.2.2.1.2. The laboratory's name, mailing address, and phone number;

8.27.2.2.1.3. The quantity of the sample;

8.27.2.2.1.4. The date of shipment; and

8.27.2.2.1.5. A description of the sample.

8.27.2.2.2. Package the sample so that it does not leak, spill, or vaporize from its packaging.

8.27.2.2.3. This exemption does not apply if the laboratory determines that the waste is hazardous but the laboratory is no longer meeting any of the conditions stated in 8.27.1 above.

8.28. **Satellite Waste or Laboratory Satellite Waste** - Hazardous waste generated by Pace that is at or near any point of generation and under the control of the operator. Satellite accumulation provisions allow generators to accumulate up to 55 gallons of hazardous waste (or 1 quart of acute hazardous waste) in containers without starting the storage clock as described in Section 3.4.

8.29. **Satellite Waste Container** - Any portable device used to accumulate laboratory generated waste prior to transfer to the hazardous waste storage area.

8.30. **Small Quantity Generator (SQG)** - A generator who produces no more than 1000 kilograms of hazardous waste (or a total of 1000 kilograms of any residue or contaminated soil, waste or other

debris resulting from the cleanup of a spill, into or on any land or water, or any acute hazardous waste) in a calendar month. The total amount of hazardous waste which may be accumulated on-site is 6000 kilograms.

8.31. **TSDF** - A Treatment/Storage/Disposal Facility.

8.32. **Universal Waste** – Commonly used items that are hazardous but can be recycled. These include fluorescent lights, computer monitors, etc.

8.33. **Waste Stream** - The generic profile of chemical and physical properties that satellite wastes exhibit.

8.34. **MMSD** – Madison Metropolitan Sewerage District is the regulatory authority in control of treating waste water discharged from the fixed base laboratory.

8.35. **WDNR** – The Wisconsin Department of Natural Resources is the regulatory authority enforcing EPA statutes in Wisconsin as well as Wisconsin state statutes related to disposal practices and waste management.

8.36. **Clean Sweep** – A facility operated by Dane County that receives hazardous materials from residents and businesses operating as very small quantity generators (VSQG).

9. Equipment and Supplies (Including Computer Hardware and Software)

The following equipment is mandatory under RCRA guidelines unless otherwise denoted. Periodic review (not to exceed monthly) of availability of equipment and supplies below should be conducted to maintain an adequate and viable supply.

9.1. **Chemical Spill Control Neutralizers**: The waste room stores three types of bulk dry spill neutralizers: solvent, acid and base. They may be utilized by placing the dry neutralizer onto a liquid chemical spill. Neutralization is indicated by a prevalent color change.

9.2. **Communication Device**: Required for emergency notification of spill, fire, etc.

9.3. **Drums**: Common types of waste drums used for storing and shipping hazardous wastes are polyethylene, steel-polyethylene lined, and steel. Sizes are typically 5gal, 15gal, 30gal, and 55gal. Drums used for liquids typically are closed top with an opening to pour the solvent through a funnel, while drums used for solids or lab packs are open-top.

9.4. **Emergency Drench Shower**: Shower should deliver water approximately twenty gallons per minute with a non-interruptible flow. It may be turned on by pulling the shower handle down. It may be turned off by pushing the handle back to the 'off' position.

9.5. **Emergency Eye Wash**: Eye wash should deliver water approximately 0.4 gallons per minute with a non-interruptible flow. It may be turned on by pushing the eye wash lever forward. It may be turned off by pulling the lever back to the 'off' position.

9.6. **Emergency Lighting** (as needed): The waste room is outfitted with emergency lighting that goes on if power fails.

9.7. **Exit Signs** (as needed): Exit signs are provided on all waste room doors. These signs are self-illuminating.

9.8. **Fire Alarm Pull Station**: A fire alarm pull station must be in close proximity to the hazardous waste room. The alarm may be activated by pulling the switch. Other alarm systems may be utilized as long as all personnel are trained on the procedures.

9.9. **Fire Extinguisher**: An extinguisher with a rating appropriate to the waste being stored in the area must be in close proximity to the hazardous waste room.

9.10. **Labels**: A multitude of labels are provided to ensure compliant labeling. They may be purchased or prepared manually.

9.11. **Liquid Chemical Neutralizers**: Liquid chemical neutralizers (base and acid) may be used to neutralize a contained hazardous liquid. This may be done by slowly adding the neutralizer to the liquid.

9.12. **pH Strips**: Wide range pH strips may be used to determine the pH of a liquid being neutralized.

9.13. **Spill Control Pads**: Spill pads are used to soak up hazardous liquids. They do not neutralize spills. They are especially effective for cleaning up oily materials. Various pads are available for aqueous and petroleum based liquids.

9.14. **Spill Control Pillows**: Spill pillows may be used to soak up large amounts of liquid chemical spills. No neutralization occurs.

9.15. **Spill Dikes**: vary depending on the size and type of room: Their purpose is to encircle a spill, barring the spread of a hazardous chemical. They will also absorb liquids, but do not neutralize spills.

10. Reagents and Standards

10.1. Not applicable to this SOP.

11. Calibration and Standardization

11.1. Not applicable to this SOP.

12. Procedure

12.1. All Pace facilities that generate hazardous waste must have a Generator's US EPA Identification Number. The ID number is obtained through the applicable EPA region's office by completing EPA form 8700-12, and must be completed before generating any hazardous waste.

12.1.1. Pace only utilizes transporters and treatment, storage, or disposal facilities (TSDFs) that have EPA identification numbers for hazardous waste handling and meet the TSDF transfer requirements.

12.1.2. A new ID number is necessary when changing locations as the number is tied to the facility address.

12.1.3. This facility's US EPA Identification Number is WIR000161547.

12.2. The laboratory generates wastes originating from several source types: materials and chemicals used to prepare and analyze samples (e.g., solvents, acids), unconsumed liquid and solid samples, certain types of batteries, mercury from lamps and broken thermometers and automobile waste. Unconsumed samples may include laboratory-contaminated sample residue (both liquid and soil) generated as part of digestion, extraction, etc., procedures used to prepare samples for analysis.

12.2.1. This facility is classified as a Conditionally Exempt Small Quantity Generator (VSQG).

12.3. Hazardous waste classification is the most critical step in establishing an effective, compliant waste-handling program. Laboratory wastes are classified using the criteria set forth under RCRA for ascertaining non-hazardous versus hazardous status, and this criterion is listed in the definition of hazardous waste in 8.20.

12.4. The following are the waste streams resulting from materials and chemicals used in the laboratory operation. Applicable information for each is given pertaining to packing, labeling, or listing on a manifest. A description of how the wastes are created, and the preferred method of final disposal for each, is included. The overriding principle in hazardous waste classification is application of a conservative formula based on all known or suspected hazards related to a waste material. While this formula may result in some materials being disposed as hazardous when in fact, they are non-hazardous (e.g., false positive), the formula will not be compromised in the interest of reducing the amount of waste produced. This will minimize any risk of a material being disposed of erroneously as non-hazardous when it, by definition, is a hazardous waste.

12.4.1. **Corrosive waste** is generated in the majority of the departments in the laboratory. This waste stream consists primarily of spent or excess aqueous reagent solutions generated from preservatives, acid digestions of metals, impinger solutions or other corrosive solutions generated in the course of analysis. The predominant corrosives include hydrochloric acid, nitric acid and sulfuric acid, but corrosives also include bases. Varying concentrations of metals may be present dependent upon the composition of the reagents added. This waste stream only has the hazardous quality of being corrosive; therefore, if a waste has any additional hazardous waste quality (e.g., Toxic or Ignitable) it cannot be mixed with this stream. This stream is most commonly treated onsite.

Corrosive Waste	
DOT Shipping Name	RQ Waste, Corrosive Liquid, N.O.S (i.e. corrosive material)
EPA Waste #	D002
Container	Various containers
Average pH	<2.0, >12.5
Disposal Method	Treatment by Neutralization
Label	Corrosive

12.4.2. **The Flammable Waste Stream** consists of flammable solvents from various processes across the lab, excluding chlorinated or hazardous materials. This waste is generated during self-contained soil extractions.

Flammable Waste	
DOT Shipping Name	RQ, Waste Flammable liquids, N.O.S. (Acetone, Methanol), Class 3, UN1993, PG
EPA Waste #	D001, F003
Container	Sample extract containers
Average pH	6-8
Disposal Method	Fuel blending
Label	Flammable

12.4.3. The **Chlorinated Waste Stream** consists primarily of methylene chloride with a very small amount of other organic solvents derived from extraction procedures performed on samples and from rinsing glassware. As a best practice, effort is made to have this waste stream as pure as possible in order to offer for recycling.

Chlorinated Solvents	
DOT Shipping Name	RQ Hazardous Waste, Toxic Liquid, N.O.S (i.e. dichloromethane, acetone, methanol)
EPA Hazard Codes	U080, F001/F002
Container	Sample extract containers
Average pH	7.0
Disposal Method	Clean Sweep
Label	Toxic, Chlorinated

12.4.4. **PCB Sample Extracts** are specific wastes that result from PCB analysis. This waste comes from the preparation PCB samples and may include vials and other disposable glassware that is used in the PCB extraction process.

PCB Sample Extracts	
DOT Shipping Name	Waste, Corrosive Liquids, Oxidizing N.O.S. (sulfuric acid)
EPA Waste #	D001, D002
Container	Sample extract containers
Average pH	<2
Disposal Method	Multiple treatments
Label	Corrosive

12.4.5. **Chromerge Waste** Chromic acid glassware washing solution

Chromerge Waste	
DOT Shipping Name	Waste Chromic acid solution, Class 8, UN1755, PG II
EPA Waste #	D002, D007
Container	Amber glass bottle
Average pH	<2
Disposal Method	Treatment
Label	Corrosive

12.4.6. **Non-Regulated Soil Samples** Non-Regulated Soil Samples are materials that are not hazardous by either RCRA or USDA.

Non-Regulated Soil Samples	
DOT Shipping Name	Non-DOT Regulated

	Material
EPA Waste #	
Container	4oz. glass jars
Average pH	NA
Disposal Method	Landfill
Label	None

12.4.7. **Methanol Soil Samples** Methanol soils are materials that are not hazardous either by RCRA or USDA, except that methanol is not poured off or evaporated in this waste stream.

Methanol Soil Samples	
DOT Shipping Name	RQ, Waste Flammable Liquids, N.O.S. (Methanol, Soil), Class 3, UN1993. PG II (D001)
EPA Waste #	D001, F003
Container	40mL VOA vials
Average pH	~7
Disposal Method	Treatment/Landfill
Label	Flammable

12.4.8. **RCRA Hazardous Samples (Solid)** RCRA Hazardous Samples are lab samples classified as hazardous by any RCRA regulation. The majority of these samples are TSCA level PCBs (>50ppm).

RCRA Hazardous Samples (Solid)	
DOT Shipping Name	Hazardous Waste, Solid, N.O.S.
EPA Waste #	D001, D002, D004, D005, D006, D007, D008, D010, D011, D012, D013, D014, D015, D016, D017, D018, D019, D020, D021, D022, D023, D024, D025, D026, D027, D028, D029, D030, D031, D032, D033, D034, D035, D036, D037, D038, D039, D040, D041, D042, D043
Container	Sealed plastic pails
Average pH	NA
Disposal Method	Incineration
Label	Corrosive, Flammable, Toxic

12.4.9. **USDA – APHIS Regulated Soil** USDA – APHIS (Foreign) Regulated Soil consists of unused regulated soils.

USDA-APHIS Regulated Soil	
DOT Shipping Name	Non-DOT Regulated Material
EPA Waste #	
Container	4oz. glass jars
Average pH	NA
Disposal Method	Incineration
Label	None

12.4.10. **RCRA Hazardous Samples (Liquid)** Lab samples hazardous by any RCRA regulation. Samples may default to this waste stream if the contents are unknown or other concerns are present (oily, stinky, unlabeled).

RCRA Hazardous Samples (Liquid)	
DOT Shipping Name	Hazardous Waste, liquid, N.O.S.
EPA Waste #	D001, D002, D004, D005, D006, D007, D008, D009, D010, D011, D012, D013, D014, D015, D016, D016, D017, D018, D019, D020, D021, D022, D023, D024, D025, D026, D027, D028, D029, D030, D031, D032, D033, D034, D035, D036, D037, D038, D039, D040, D041, D042, D043
Container	Various containers
Average pH	Varies
Disposal Method	Treatment
Label	Corrosive, Flammable, Toxic

12.5. Some waste can become complicated when attempting to classify as non-hazardous or hazardous due to the list of hazardous constituents contained in sections 40 CFR 261.30-261.35 including a majority of analytes of interest routinely analyzed in Pace laboratories. Definitions have been established for each of the F, K, P, and U lists covering hazardous waste originating from non-specific sources, specific sources and discarded commercial chemical products, off-specification species, container residues, and spill residues. The application of listed hazardous wastes and substances is intended for manufacturing processes involving pure products, by-products, wastes generated as part of the production process and cleanup of materials contaminated from a spill of the listed commercial chemical product or manufacturing chemical intermediate. See Attachment I for common F-listed wastes.

12.5.1. Hazardous waste classification of unconsumed samples by listed hazardous waste criteria is not commonly applied in laboratory operations. Examples of sample types which would be identified as listed hazardous wastes include the following:

12.5.1.1. Samples containing 5% or more (by volume) of halogenated and non-halogenated "spent solvents:" (e.g., drum sample with > 10% TCE);

12.5.1.2. Pure product and two phase solution samples containing a listed chemical product or manufacturing intermediate (e.g., drum sample);

12.5.1.3. Samples from specific sources listed in section 261.32 (e.g., bottom sediment sludge from the treatment of wastewaters from wood-preserving processes that use creosote and/or pentachlorophenol - K001);

12.5.1.4. Samples representing any residue or contaminated soil, water or other debris resulting from the cleanup of a spill into or on any land or water of any commercial chemical product or manufacturing chemical intermediate having a generic name listed in section 261.33, or any residue or contaminated soil, water or other debris resulting from the cleanup of a spill, into or on any land or water, of any off-specification chemical product and manufacturing chemical intermediate which, if it met specifications, would have the generic name listed in section 261.33.

12.5.2. For the wastes listed in 12.5.1.1 and 12.5.1.2, disposal can be achieved by individually lab packing them or combining with other compatible hazardous wastes.

12.5.3. The remaining two sample types in 12.5.1.3 and 12.5.1.4 would also require lab packing for disposal. However, it is important to note that in order for the laboratory to ascertain that the samples were derived from a specific listed source or from a spill of a listed chemical, they must be so informed by the industrial concern or lead agency (e.g., EPA, state regulators) submitting the sample for analysis. If a water or soil sample contains a listed hazardous waste substance whose origin is unknown or uncertain to the lead agency, then that sample is not classified as a listed hazardous waste. Rather in this case, determination of a hazardous waste classification can only be obtained by the waste exhibiting a characteristic of hazardous waste (e.g., ignitability, corrosivity, reactivity).

12.5.4. Due to the fact that the majority of samples analyzed by Pace do not meet the well-defined criteria for identifying "listed" hazardous waste, disposal classification of unconsumed samples will be based upon characteristics of hazardous waste:

12.5.4.1. Non-Hazardous - Analysis results indicate an absence of contaminants; unless contaminants listed under the hazardous disposal categories are parts of the requested sample analysis.

12.5.4.2. Hazardous – Analysis results indicate presence of contaminants (Attachment III) or sample analysis requires hazardous materials and contaminants. Samples in this category are segregated from others and disposed of as hazardous according to laboratory procedures.

12.5.4.3. PCB Waste - Generated exclusively by samples contaminated with greater than trace levels of polychlorinated biphenyls (≥ 50ppm). Samples containing 50ppm (total) or higher of PCBs must be segregated and disposed of as PCB waste.

12.5.4.4. Waste Oil/Paint - Samples which are predominantly of an oil matrix (e.g., highly viscous organic liquid) or paint (solvent and pigment blend) are segregated and disposed in a separate container. Though these samples are defined as nonhazardous, oil samples are a special case and never disposed as nonhazardous. Note: Bottle caps and liners do not typically contain sample residuals and can be disposed of directly through the non-hazardous building refuse.

12.5.5. USDA-APHIS-PPQ Regulated Soils (Regulated Soils) are a special case of sample strictly controlled under quarantine regulations 7 CFR 330 because they can readily provide a pathway

for a variety of dangerous organisms throughout the United States. The movement of soil into the United States from foreign sources and from certain regulated areas within the continental U.S. is restricted unless permitted by APHIS under specific conditions and safeguards.

12.5.5.1. Any laboratory that plans to handle Regulated Soils must have an approved Soil Permit or Regulated Soil Agreement from USDA-APHIS-PPQ. See the updated revision of *Regulated Soil Handling SOT-ALL-S-003* for all information regarding the handling of these materials.

12.5.6. Though Pace is obligated to ensure nonhazardous discharge complies with requirements set by applicable publicly owned treatment works (POTW), Pace is not obligated to run every available analysis on every sample for proper waste classification. Consequently, samples are characterized according to the preservatives added, the requested analytical testing data, and any knowledge of the sample provided by the client. When sample analysis is canceled/not completed, those untested samples only need be characterized by the preservatives added and any knowledge of the sample that is obtained by the client.

12.6. Consolidation of wastes from the laboratory proceeds via two distinct routes covering either laboratory-generated hazardous wastes or excess unconsumed samples.

12.6.1. Laboratory Accumulation and Satellite Waste Containers

12.6.1.1. Waste materials from routine lab procedures are collected in containers of appropriate construction, placed in convenient locations at the point of generation. Under RCRA guidelines, these are defined as satellite containers.

12.6.1.2. The amount of hazardous waste stored in the laboratory at the individual satellite areas cannot exceed 55 gallons (liquid) or 550 lbs (solid) per waste stream, for non-acute hazardous waste.

12.6.1.3. Satellite waste containers must be labeled in accordance with all regulations, including:

12.6.1.3.1. Designation of the contents to be hazardous waste with the words "Hazardous Waste" clearly legible.

12.6.1.3.2. The waste stream description (e.g., acid waste).

12.6.1.3.3. A hazard label (e.g., corrosive).

12.6.1.4. The satellite containers must be maintained such that evolution of chemical vapors is precluded. This requires that the container be closed at all times, except when adding or emptying hazardous waste to and from the container.

12.6.1.5. The most critical point in the waste handling system is when a person (e.g., analyst, technician) places a waste material into a satellite container. Here, the characteristics or listing of the waste and the waste stream must both be known to match. For this reason, only material from approved procedures should be placed in the compatible satellite containers. All materials from experimental procedures, unknown or out of the ordinary sources, or from spill cleanups must be characterized and described to the Hazardous Waste Coordinator, who determines the proper method of disposal.

12.6.1.6. Full satellite containers must be transferred to the proper accumulation drum within 3 calendar days. Lab collection containers must not be filled to the top of the opening. Space must be left to prevent splashing of hazardous material when containers are emptied and to allow for expansion and contraction within the drum during transport.

12.6.1.7. Satellite containers for liquid hazardous waste must have secondary containment made of material that could successfully contain the entire satellite container's contents.

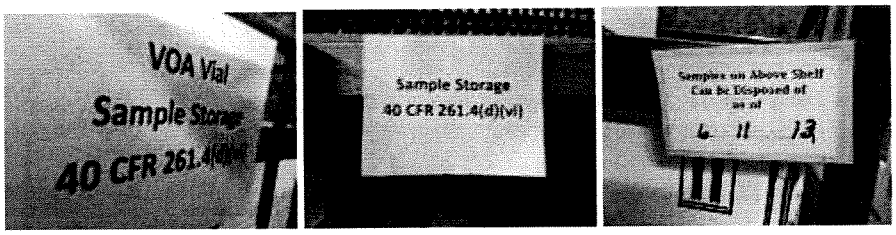
12.6.2. Unconsumed Sample Disposal

12.6.2.1. Client samples are stored on-site for a defined period of time after the final analytical report is generated and prior to sample disposal. The purpose of sample storage is to provide the client time to review the analytical report and determine if the samples require additional testing or need to be returned to the client. Samples are not considered a waste during this time according to 40 CFR 261.4(d)(vi).

12.6.2.1.1. The default sample storage time is **"365 days from Login."** Therefore, the maximum sample storage time is one (1) year from the login date. Other sample storage hold times may be assigned for specific contractual requirements.

12.6.2.1.2. In the fixed base laboratory, sample management information is documented in an excel file called 0Disposal.xxxx where xxxx is the current year. (Ex. 0Disposal.2017)

12.6.2.1.3. During sample storage, the process and sample status must be obvious to employees, customers and auditors. This transparency is imperative to ensure samples are considered active test specimens to be retained until they are categorized as a waste for disposal.



12.6.2.2. Samples which cannot be returned to the client for disposal are characterized according to section 12.4. Samples are characterized by one of three methods:

12.6.2.2.1. Analytical results are evaluated against characterization criteria established for the sample waste stream. The samples which exhibit waste characteristics as previously outlined are segregated and denoted per laboratory/facility policies. The completed form is then forwarded to the Hazardous Waste Coordinator, who in turn uses the information to coordinate removal of unconsumed samples from active sample storage by the log-in staff. OR:

12.6.2.2.2. Samples are scanned out of EPIC Pro (LIMS) as RCRA nonhazardous to be disposed as the waste stream is normally handled unless the sample tested over RCRA limits, in which the LIMS will prompt the employee that the sample is scheduled for Hazardous disposal, and is segregated from the nonhazardous samples. OR:

12.6.2.2.3. Samples of a certain type are all "assumed" to be hazardous, and all are placed into an accumulation drum with all required RCRA labeling for that waste stream.

12.7. Transferring Satellite Waste to the Waste Storage/Accumulation Area

12.7.1. All transfers of satellite waste to waste drums must be made by the Hazardous Waste Coordinator or designated, trained personnel. When a satellite waste container is full, the Hazardous Waste Coordinator, or designee must be notified. Regular disposal events may be scheduled to dispose satellite waste on a continuous basis.

12.7.2. Find the correct waste drum by referring to the Hazardous Waste placard and hazard label. Mixing solvents that are not compatible could result in a hazardous reaction.

12.7.3. Ensure there is enough capacity in the drum to hold all the content that will be dispensed.

12.7.4. Check to make sure there is a ground connection before opening a solvent waste drum.

12.7.5. Open and slowly pour the contents of the satellite container into the proper waste drum using an appropriate solvent resistant funnel.

12.7.6. Replace the cap on the bunghole and carefully screw the cap on but do not tighten the cap.

12.8. Unconsumed Soil Samples

12.8.1. In the mobile labs, samples are returned to the client.

12.8.2. In the fixed base lab, samples are pulled from storage and disposed of according to the sample disposal Excel document (0Disposal.xxxx).

12.8.2.1.1. Water samples are disposed no sooner than one month from receipt. Water samples are disposed by dumping them down the drain through a carbon filter. The cold water faucet is left running at a medium flow to flush the drain during the disposal and for at least 10 minutes following disposal.

12.8.2.1.2. Soil samples are disposed no sooner than six months from receipt. After keeping soil samples in cold storage for at least a month, they can be moved to warm storage. These samples are organized into labeled boxes and stored offsite.

12.8.2.1.3. Before being disposed, samples are queried in LIMS (Promium Element) and any samples with detections above the appropriate hazard level are segregated. This is done in Sample Control/Disposal. Samples with detections are denoted in the 'Hazard Info' column.

12.8.2.1.4. Soil samples are composited and tested before disposal. The sample to be tested is obtained by taking a subsample from five random jars in each box to be disposed. The subsamples are then composited and tested for 8141, 8151, and 8081 pesticides and/or specific landfill testing requirements.

12.8.3. Until soil is sterilized per the instructions in SOP-MAD-S-003, the foreign or USDA regulated domestic soil must be stored, processed, tested, and heat treated in the laboratory. The laboratory must be restricted to access of authorized employees only. Information regarding the handling of foreign or USDA regulated domestic soil is available in SOP-MAD-S-003.

12.9. Elementary Neutralization

12.9.1. Dilute corrosive solutions (e.g., preserved metals samples) which do not exhibit any hazardous characteristics other than being corrosive, may be neutralized. Elementary neutralization is exempt from RCRA permitting requirements for on-site hazardous waste treatment. While exempt under RCRA guidelines, before utilizing this practice to reduce off-site treatment or disposal of wastes, local pretreatment and discharge standards must be met for publicly owned treatment works (POTW).

12.9.2. The discharges listed below are prohibited under the National Pretreatment Standards and Prohibited Discharge Standards:

12.9.2.1. Pollutants causing fire or explosion (waste with a flashpoint < 60°C);

12.9.2.2. Corrosive wastes with pH less than 2 or greater than 12.5;

12.9.2.3. Solid or viscous pollutants that could potentially block the system;

12.9.2.4. Oxygen-demanding pollutants;

12.9.2.5. Wastes which generate toxic gases.

12.9.3. Neutralization Procedure

12.9.3.1. Obtain the 5-gallon bucket used for neutralizing wastes and place it in the sink, if possible.

12.9.3.2. Slowly pour waste to be neutralized into bucket up to the fill line indicated in black marker.

12.9.3.3. Using a wide-range pH test strip or pH meter, measure the pH of the waste material.

12.9.3.4. Gradually add neutralizing agent, 10N NaOH to raise the pH and nitric acid to lower the pH, to the 5-gallon bucket containing waste.

12.9.3.5. Between each addition of neutralizing reagent, stir the solution with a glass or wooden stirring rod to ensure thorough mixing.

12.9.3.6. Test the pH again with a pH test strip or with a pH meter.

NOTE: Initially the neutralizing reagent may be added in larger increments (approximately 100 mL). As the pH gets closer to neutral, add smaller quantities so as not to overshoot the desired pH.

12.9.3.7. Repeat steps 12.9.3.4 to 12.9.3.6 until a pH of 5-9 is obtained.

NOTE: Should the pH range of 5-9 be inadvertently exceeded, simply add the other neutralizing reagent to further adjust the pH.

12.9.3.8. Once a pH in the desired range is obtained, the waste can be disposed of down the drain with cold running water.

12.10. Waste Storage Container Requirements

12.10.1. Drums in the hazardous waste storage area are labeled consistent with both DOT and EPA regulations concerning hazardous materials and wastes (see Attachment IV for example of label).

12.10.2. Labels must be easily visible and legible (e.g., a drum must not be labeled and then placed in such a way that the label cannot be seen).

12.10.3. The Accumulation Start Date must be recorded on the drum. The date should reflect the first time waste was added to the drum and not the date when the waste was generated in the laboratory.

12.10.3.1. Once a waste is removed from the point of generation to a hazardous waste staging area, the clock is started for storage time prior to disposal.

12.10.3.2. Drums must be picked up by TSDF for disposal before accumulation time exceeds RCRA requirement for lab's generator status (see section 3.4).

12.10.4. The hazardous waste staging room must be arranged in such a fashion to assure direct access pathways in the event of foreseeable emergency and for safe waste transfer. A minimum aisle space of three feet must be maintained at all times to access hazardous waste containers.

12.10.5. All hazardous waste drums and containers must be securely closed when not in use. All volatile and flammable hazardous waste liquid containers must be securely grounded at all times. Drums

containing these liquids should also be manipulated with non-sparking tools and fitted with a drum venting bung, to assure that excess pressure build-ups are safely released.

12.10.6. All liquid waste stream containers must be provided with secondary containment devices. Such containment devices must be made of materials compatible with each waste, and they must be free of leaks. The waste storage room may act as secondary containment as long as the room has been constructed to safely and effectively contain a hazardous waste spill.

12.10.6.1. Secondary containers must exceed the total volume of the largest container stored in each containment device for indoor storage.

12.10.7. Compatibility of wastes must be considered in arranging storage areas. For example, acid waste should never be stored adjacent to basic waste, particularly cyanide wastes. Further examples are outlined in 40 CFR 264, Appendix V.

12.10.8. The hazardous waste staging area is controlled so unauthorized personnel are not able to access the room or contents.

12.10.9. The maximum volume of acutely hazardous waste (e.g., P-listed wastes) that can be accumulated in the laboratory is one quart. The volumetric measurement of one quart is based upon container size in which the waste is stored and not the actual amount (volume) of waste present. An example of how this one quart limit can inadvertently be exceeded involves the disposal of a neat standard of 2,4-dinitrophenol into a one gallon bottle. While the neat standard itself may only constitute 1-2mL, the volume as defined under RCRA would be one gallon, thus the laboratory would be out of compliance.

12.11. Waste Documentation and Reporting

12.11.1. All drums containing hazardous waste are recorded in a logbook or database. The information contained in this log is useful when filling out EPA biennial reports and for retaining an accurate description of how much waste has been accumulated. The following information is entered into the logbook/database;

12.11.1.1. The drum number;

12.11.1.2. The date filling the drum was started;

12.11.1.3. The drum capacity (e.g., 55-gallon, etc.);

12.11.1.4. The manifest number associated with the drum's disposal.

12.11.2. The following hazardous waste records must be maintained a minimum of five years and should be retained indefinitely:

12.11.2.1. Drum tracking logs;

12.11.2.2. Sample Reports;

12.11.2.3. Sample disposal information and waste records on computer disc;

12.11.2.4. Analytical records relating to sample waste stream profiling and characterization;

12.11.2.5. Labpack inventory logs;

12.11.2.6. Biennial Reports, Exception Reports, or other reports filed for compliance reasons;

12.11.2.7. Records related to unresolved enforcement action must be retained indefinitely until such a time that the matter is resolved;

12.11.2.8. Facility Certificates of Destruction or Recycling.

12.11.3. A Waste Manifest is the documentation form that must accompany all shipments of hazardous waste while in transit.

12.11.3.1. A Hazardous Waste Manifest Cover Sheet (*F-ALL-W-DRAFT-rev.00 or local replacement*) will be utilized to ensure waste transfer from generator to TSDF fulfills all legal requirements.

12.11.3.2. The manifest is to be signed and dated by a DOT trained Pace employee responsible for the shipment and the transporter. The transporter will leave 2-3 of these "two-signature page" copies of the manifest.

12.11.3.3. Within 30 days of the waste leaving the site, save copies/pages to the waste management folder.

12.11.3.4. Within 35 days you will receive a three-signature page (generator, transporter, facility) showing the waste reached its intended destination. The waste coordinator is responsible to ensure it is properly completed.

12.11.3.4.1. If you do not receive the three signature page within 35 days of shipment, call the facility to find out why you have not received it. If you do not receive the three-signature page within 60 days you must file an exception report with WI-DNR.

12.11.3.5. All manifests must be kept for a minimum of three years.

12.11.4. The central accumulation staging room and satellite waste containers must have documented inspection as part of the monthly laboratory inspection. The inspections should ensure all regulations are obeyed; see sections 12.10 and 12.6.1 for accumulation storage and satellite rules.

12.11.4.1. A record of the inspections must be kept in an inspection log or summary.

12.11.4.2. Records must be maintained for at least three years from the date of inspection. At a minimum, the records must indicate:

12.11.4.2.1. The date and time of the inspection;

12.11.4.2.2. The name and signature of the inspector (typically will be Hazardous Waste Coordinator);

12.11.4.2.3. A notation of the observations made (can be in a check-off format, e.g., fire extinguisher: charged _X_ requires recharging __);

12.11.4.2.4. The date and nature of any repairs or other remedial actions.

12.11.5. Annual Generation Reports are required to be filed for SQGs and LQGs; at this time this facility is a VSQG and does not have any annual reporting requirements.

13. Quality Control

13.1. Not applicable to this SOP.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. The analyst must read and understand this procedure with written documentation maintained in his/her training file.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A formal safety plan is also available to all employees. Gloves and eye protection should be routinely worn when handling reagents or samples. When qualified employees are transferring wastes, additional protection such as goggles, face shield, and lab apron are recommended.

22.2. Safety Data Sheets are located in a central location and should be consulted prior to handling samples and standards.

22.3. A hazard assessment must be completed per S-ALL-S-001 ***Hazard Assessment*** for waste areas to ensure proper PPE are utilized.

23. Waste Management

23.1. Not applicable to this SOP.

24. Pollution Prevention

24.1. Not applicable to this SOP.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. 40CFR261-268, Code of Federal Regulations Chapter 1, Subchapter I – Solid Waste.

25.3. 29CFR171-174, Code of Federal Regulations, Transportation.

25.4. Federal Plant Pest Regulations, Part 330.

25.5. Department of Defense (DoD) Quality Systems Manual- most current version.

25.6. Is Your Waste Hazardous?, WDNR Publication WA 1152, Rev. 2007, http://dnr.wi.gov/files/PDF/pubs/wa/wa1152.pdf

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: RCRA Requirements for Labs as a Function of Generator Status.

26.2. Attachment II: Hazard Codes for Common F-List Wastes (solvents).

26.3. Attachment III: TCLP Contaminant List with Concentration Limits.

26.4. Attachment IV: Hazardous Waste Label for Accumulation Drum (example).

26.5. Attachment V: Satellite Container Inspection Form (example).

26.6. Attachment VI: Waste Accumulation Room Inspection Form (example).

26.7. Attachment VII: Hazardous Waste Manifest Cover Sheet.

27. Revisions

Document Number	Reason for Change	Date
SOT-ALL-W-002-rev.05	Section 8.20.2.4: Added definition of Toxic. Section 12.5.5: Added more detail to USDA Regulated Soils. Section 12.6.2.1: Defined sample storage hold times. Added Section 12.11.3.1 and Attachment VIII– Hazardous Waste Manifest Cover Sheet info. Clarity added throughout that liquid waste containers need secondary containment, not all waste containers. Referenced sections and regulations corrected.	14Nov2013

SOT-ALL-W-002-rev.06	Section 8.27: reorganized for clarity and fixed reference section errors. Section 8.28: updated definition. Sections 12.5.5 and 12.5.5.1: updated to reference new, local version of Regulated Soil Handling SOT. Section 12.5.6: clarification added that Pace labs must obey discharge rules. Section 12.6.2.2.4: added red text paragraph regarding removal of sample labels for ESI clients. Removed old Attachment IV (soil regulations map) because it was outdated and this topic is covered in Regulated Soil SOP.	24Mar2015

Attachment I: RCRA Requirements for Labs as a Function of Generator Status

Requirement (40CFR)	CESQG	SQG	LQG
Waste Determination (262.11)	Applicable	Applicable	Applicable
Generation Rate Limits (261.5 and 262.34)	<100 kg/mo	100-1,000 kg/mo	1,000 kg/mo or greater
Accumulation Quantity Limit w/o Permit (261.5 and 262.34)	Not to exceed 1,000 kg at any time. Not to exceed 1 kg acute at any time	not to exceed 6,000 kg at any time	No limit
Accumulation Time (261.5 and 262.34)	No limit	180 days or 270 if waste is to be transported over 200 miles.	90 days
EPA ID Number (262.12)	Not required***; possible state requirement	Required	Required
Mark Containers with Start Date (262.34)	Not applicable	Applicable	Applicable
Mark Containers "Hazardous Waste" (262.34(a))	Not applicable	Applicable	Applicable
Air Emission Standards 40 CFR 265 Subpart CC	Not applicable	Not applicable	Applicable
Satellite Accumulation (262.34(c))	Not applicable	Applicable	Applicable
Use Manifests (262, Subpart B)	Not required; possible state requirement	Required	Required
Exception Reporting (262.42)	Not required	Required after 60 days. No TSDF notification requirement.	Required after 45 days. Notification of TSDF within 35 days.

Requirement (40CFR)	CESQG	SQG	LQG
Biennial Report (262.41)	Not required	Not required; possible state requirement	Required
Contingency Plan (265, Subpart D)	Not required, but OSHA (29 CFR 1910.38) requires emergency planning	Basic planning required in accordance with the standards in 262.34(d)(4) and (5) and 265, Subpart C as well as OSHA regulations	Full written plan in accordance with 265 Subpart D, is required by 262.34(a)(4) and OSHA regulations
RCRA Personnel Training (262.34 and 265.16)	Not required, but recommended	Basic training required by 262.34(d)(5)(iii)	Full compliance with the training requirements in 265.16 is required by 262.34(a)(4)
Storage Requirements (without permit) (262.34 and 265)	None, but OSHA regulations under 29 CFR 1910, Subparts H and N, apply, particularly 29 CFR 1910.106	Compliance with technical standards in Part 265, Subparts I and J; for containers and tanks is required by 262.34(d)(2) and (3) and OSHA regulations	Compliance with technical standards in Part 265, Subparts I, J, W, and DD, is required by 262.34(a)(1) and OSHA regulations
Recordkeeping Requirements (262.40)	Waste determinations and generation log required (notification of regulated waste activity, training records, manifests, and land disposal restriction notifications recommended)	Notification of regulated waste activity, waste determinations, generation log, manifests, land disposal restriction notifications, exception reports, and correspondence with local emergency responders (written contingency plan, weekly container inspection & periodic equipment maintenance logs, and RCRA training records recommended)	Notification of regulated waste activity, waste determinations, generation log, manifests, land disposal restriction notifications, exception reports, biennial reports, correspondence with local emergency responders, RCRA training records, and written contingency plan required (weekly container inspection is required & periodic equipment maintenance logs is recommended)
Waste "Designated Facility"	State-approved or RCRA permitted facility or legitimate recycler	RCRA-permitted facility or legitimate recycler	RCRA-permitted facility or legitimate recycler
Land Disposal Restrictions (268.7)	Possible state requirement	Applicable	Applicable

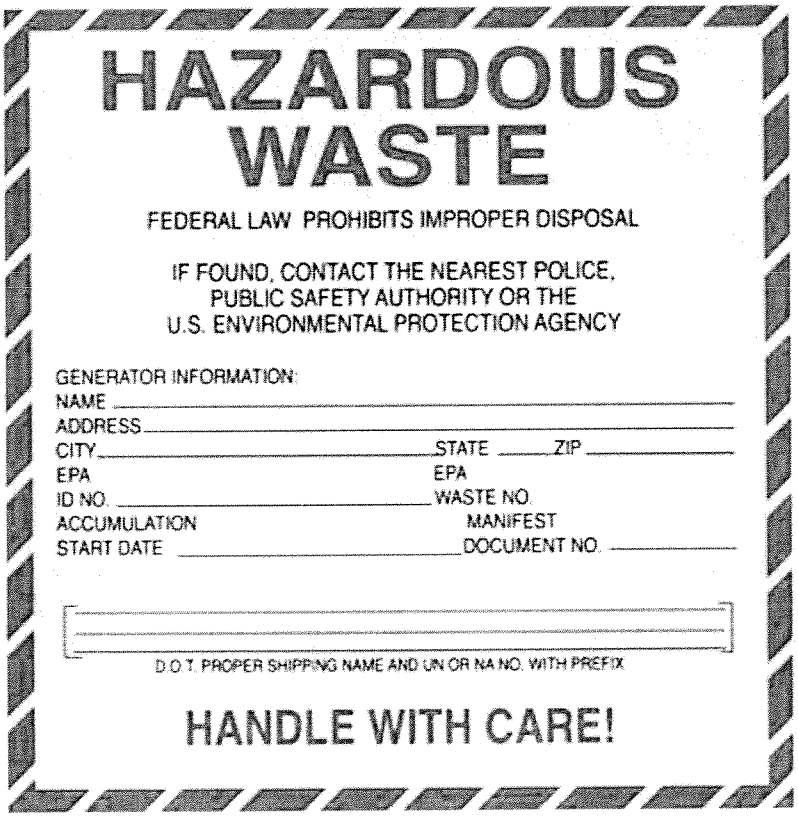
Attachment II: Common F-Listed Solvents

Waste Name	Hazardous Waste Code(s)	Waste Name	Hazardous Waste Code(s)
Acetone	F003	Methylene Chloride	F001, F002
Benzene	F005	Methyl ethyl ketone (MEK)	F005
iso-Butanol	F005	Methyl isobutyl ketone	F003
n-Butyl alcohol	F003	Nitrobenzene	F004
Carbon Disulfide	F005	2-Nitropropane	F005
Carbon Tetrachloride	F001	Orthodichlorobenzene	F002
Chlorobenzene	F002	Pyridine	F005
Chlorinated fluorocarbons (CFC)s	F001	Tetrachloroethylene	F001, F002
Cresols	F004	Toluene	F005
Cresylic acid	F004	1,1,1-Trichloroethane	F001, F002
Cyclohexanone	F003	1,1,2-Trichloeoethane	F002
2-Ethoxyethanol	F005	1,1,2-Trichloro-1,2,2-trifluoroethane	F002
Ethyl acetate	F003	Trichloroethylene	F001, F002
Ethyl benzene	F003	Trichloroflourormethane	F002
Ethyl ether	F003	Xylene	F003
Methanol	F003		

Attachment III: TCLP Contaminant List

Waste ID #	Contaminant	Conc (mg/L)
D004	Arsenic	5.0
D005	Barium	100.0
D006	Cadmium	1.0
D007	Chromium	5.0
D008	Lead	5.0
D009	Mercury	0.2
D010	Selenium	1.0
D011	Silver	5.0
D012	Endrin	0.02
D013	Lindane	0.4
D014	Methoxychlor	10.0
D015	Toxaphene	0.5
D016	2,4-D	10.0
D017	2,4,5-TP Silvex	1.0
D018	Benzene	0.5
D019	Carbon Tetrachloride	0.5
D020	Chlordane	0.03
D021	Chlorobenzene	100.0
D022	Chloroform	6.0
D023	o-Cresol	200.0
D024	m-Cresol	200.0
D025	p-Cresol	200.0
D026	Cresol	200.0
D027	1,4-Dichlorobenzene	7.5
D028	1,2-Dichloroethane	0.5
D029	1,1-Dichloroethylene	0.7
D030	2,4-Dinitrotoluene	0.13
D031	Heptachlor	0.008
D032	Hexachlorobenzene	0.13
D033	Hexachlorobutadiene	0.5
D034	Hexachloroethane	3.0
D035	Methyl ethyl ketone	200.0
D036	Nitrobenzene	2.0
D037	Pentachlorophenol	100.0
D038	Pyridine	5.0
D039	Tetrachloroethylene	0.7
D040	Trichlorethylene	0.5
D041	2,4,5-Trichlorophenol	400.0
D042	2,4,6-Trichlorophenol	2.0
D043	Vinyl Chloride	0.2

Attachment IV: Hazardous Waste Label for Accumulation Drum (Example)



HAZARDOUS WASTE

FEDERAL LAW PROHIBITS IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE,
PUBLIC SAFETY AUTHORITY OR THE
U.S. ENVIRONMENTAL PROTECTION AGENCY

GENERATOR INFORMATION:
NAME _____
ADDRESS _____
CITY _____ STATE _____ ZIP _____
EPA ID NO. _____ EPA WASTE NO.
ACCUMULATION START DATE _____ MANIFEST DOCUMENT NO. _____

D.O.T. PROPER SHIPPING NAME AND UN OR NA NO. WITH PREFIX

HANDLE WITH CARE!

Attachment V: Satellite Container Inspection Form

Waste Container ID	Clearly Labeled as "Hazardous Waste" with Waste Stream	Liquid Waste has Secondary Containment	Closed when not in use
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No
	Yes / No	Yes / No	Yes / No

If any of the above fields are a "NO", please document how the container was brought back into compliance.

Comments: _____

_____

_____

Inspector Signature _____ **Date:** _____

Reviewer Signature _____ **Date:** _____

ATTACHMENT VI: WASTE ACCUMULATION ROOM INSPECTION FORM

Containers closed when not in use	**Labels Easily Seen and Legible**	**Drums have Accumulation Start Date**	**Storage Amounts and Limits Obeyed[1]**	**Secondary Containment** for Liquid Waste	**Adequate Aisle Space**	**Available Emergency Equip. and Materials**	**Signature and Date of Inspection**	**Corrective Action for NO Answers**
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		
Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No		

1: Accumulation limits are 90 days for LQG, and 180 days for SQG. SQG may have no more than 6000kg waste at any time.

ATTACHMENT VII: HAZARDOUS WASTE MANIFEST COVER SHEET

HAZARDOUS WASTE PICKUP DATE: _____

MANIFEST NUMBER(S)	**3-SIGN. MANIFEST RETURN DATE**
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

2-SIGNATURE FORM/PICK-UP CHECKLIST

- ☐ **DRUMS REQUESTED FOR PICKUP/PRESENT MATCHES MANIFEST**
- ☐ **HAZARD CODES ARE CORRECT FOR EACH WASTE STREAM**
- ☐ **PACE REPRESENTATIVE AND TRANSPORTER SIGNATURE PRESENT**

35 DAYS FROM PICKUP DATE: _____

If the three signature page has not been received within 35 days, contact the disposal facility to determine where the shipment is and request a copy of the three signature page.

If the three signature page has not been received within 45 days, you are required to file an exception report with the local regulating authority.

ALL 3-SIGNATURE MANIFEST(S) RECEIVED DATE_____

- ☐ **FILE COVER SHEET, 3-SIG AND 2-SIG FORMS TOGETHER IN FOLDER, BINDER OR BY PAPER-CLIP. RETAIN FOR AT LEAST 3 YEARS**

WASTE COORDINATOR:_____

DATE COMPLETED:_____

P-22

Self-Contained Water Extraction (Rev.0.4; 03/28/2016; PRE-002)

SELF-CONTAINED WATER EXTRACTION

METHOD 3511

1 SCOPE AND APPLICATION

1.1 Method 3511 is a procedure for extracting selected semi-volatile organic compounds from aqueous matrices. The approach minimizes solvent usage, thereby reducing the supply costs, health and safety issues, and waste generated.

1.2 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

1.3 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2 SUMMARY OF METHOD

2.1 A measured volume of sample, usually 400 mL, at a specified pH (See Table 1), is extracted with an appropriate solvent (See Table 1) using a 500 mL amber bottle.

2.2 The extract is dried, cleaned up as necessary, and immediately ready to be analyzed by the definitive method.

3 DEFINITIONS AND ACRONYMS

3.1 There are many terms and acronyms used throughout this document. Check the definitions and acronyms sections of the Quality Manual for complete explanations.

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in the extracted ion chromatograms. Reagent blanks are analyzed with every analysis batch to monitor for potential interferences with a goal of the procedure being interference free.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the ECCS Chemical Hygiene Plan (CHP), and this document. Refer to the CHP for more detailed safety information or for information not listed in this document.

5.2 Eye protection that protects against splash and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled during this procedure.

5.3 Employees must handle glassware and equipment carefully in order to prevent injury and accidents. Any damaged or broken glassware is to be discarded or moved to the glass repair box.

5.4 ECCS maintains a Material Safety Data Sheet (MSDS) for every chemical used in the laboratory. The MSDS file is kept in the main laboratory.

6 APPARATUS AND MATERIALS

6.1 Amber glass narrow mouth bottles: 1000 mL, 500 mL or 250 mL

6.2 Vials:

6.2.1 12 mL storage vials with Teflon® lined screw cap

6.2.2 60 mL glass VOA vials

6.2.3 20 mL glass scintillation vials

6.3 Syringes: Various sizes, 10-1000 µL, Gastight

6.4 Repeater Plus with Combitips of various sizes (Eppendorf or equivalent)

6.5 Glass disposable transfer pipettes - 5 3/4" and 9" with bulbs

6.6 Assorted laboratory glassware

6.7 Optifix solvent dispensers capable of dispensing accurate volumes (EM Science or equivalent)

6.8 Wide range pH indicator paper, 1 to 14

6.9 Refrigerator capable of maintaining 4 °C

6.10 Freezer capable of maintaining temperatures below -15 °C

6.11 Fume hood capable of maintaining flow of 100 linear feet per minute

6.12 Muffle furnace capable of maintaining 400 °C

7 REAGENTS

7.1 Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

7.2 Solvents

7.2.1 Iso-octane, C_8H_{18} – pesticide quality or equivalent

7.2.2 De-ionized (DI) water, organic free

7.2.3 Hexane, C_6H_{14} – pesticide quality or equivalent

7.3 Solid Reagents

7.3.1 Sodium chloride (NaCl) - reagent grade

7.3.1.1 Sodium chloride is baked in a muffle furnace at 400 °C for a minimum of 4 hours and is stored in sealed glass containers prior to use. Label each container with the LIMS ID.

7.3.2 Sodium sulfate (Na_2SO_4) – reagent grade

7.3.2.1 Sodium sulfate is baked in a muffle furnace at 400 °C for a minimum of 4 hours and is stored in sealed glass containers prior to use. Label each container with the LIMS ID.

7.4 Acids and Bases

7.4.1 10 N sodium hydroxide (NaOH) solution – VWR Scientific

7.4.2 Concentrated sulfuric acid (H_2SO_4), EM GR grade

7.4.3 12 N H_2SO_4 - Caution: excessive heat is generated when preparing this acid.

7.4.3.1 Slowly add 333 mL of concentrated H_2SO_4 to 400 mL of de-ionized (DI) water in a 1 L volumetric flask placed in iced water. Fill to the mark with DI water and invert. Allow to cool and fill to the mark with DI again. Transfer the contents to a one liter bottle. Document the preparation in LIMS, label appropriately, and assign a 1 year expiration date.

7.5 Surrogate spiking solution

7.5.1 See Table 2 for surrogate spiking information for each definitive method.

7.6 Laboratory control sample (LCS)/matrix spike/matrix spike duplicate (MS/MSD) spiking solution

7.6.1 See Table 2 for LCS and MS/MSD spiking information for each definitive method.

8 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples should be collected in 1 L amber glass bottles with Teflon-lined caps and refrigerated at 4 °C after collection. Check the determinative method SOP for additional preservation requirements. Request one of the samples to be collected in duplicate for MS/MSD analysis.

8.2 Aqueous samples should be extracted within 7 days of collection. Store extracts in a freezer at -15 °C or below and analyze within 40 days of extraction.

9 PROCEDURE

9.1 See Table 1 for specific definitive method information.

9.2 Remove the samples to be extracted, and the surrogate and matrix spiking solutions from cold storage and allow to come to room temperature.

9.3 Prepare a minimum of one blank, LCS, and MS/MSD with each set of 20 or less water samples extracted per day. Use DI water for the blank and LCS, except for TCLP extracts, use the appropriate fluid. See Table 1 for volume used. If no sample volume is provided for MS/MSD, an LCS duplicate must be prepared with the batch.

NOTE: If a single extraction is being performed for multiple definitive methods, prepare separate samples for the LCS, MS, and MSD per each definitive method.

9.4 Glassware used during the extraction process is generally single use and can be used as is. Glassware that is not single use must be pre-rinsed with acetone or another appropriate solvent prior to use.

9.5 Transfer the appropriate volume of sample (See Table 1 for recommended volume) to the appropriate bottle (See 9.6) and add the appropriate amount of purified NaCl (See 9.6). Table 1 lists the default initial and final volumes, but LIMS will adjust the LOD and LOQ based on preparation ratio variations as long as the analyst does not change the default value in LIMS on the batch page. Record actual initial and final volumes on the preparation bench sheet in LIMS. LOD studies are conducted based on the volumes in Table 1.

9.6 Alternate sample volumes may be extracted to accommodate project specific requirements. However, the amount of NaCl added needs to be proportionate to the volume of water used.

Bottle Size	Volume	NaCl Weight
60 mL VOA	40 mL	15 g
250 mL	200 mL	70 - 75 g
500 mL	400 mL	140 – 150 g
1000 mL	800 mL	280 – 300 g

9.7 Check the pH of the samples with wide-range pH paper and, if necessary, adjust the pH to 5-9 using 12 N sulfuric acid or 10 N sodium hydroxide unless footnote indicated differently in the Initial pH column of Table 1. If adjusted, record the pH in LIMS. Also record the amount of acid or base used.

NOTE: The pH check is required for 8015, 8081, and 8082 but pH adjustment is not.

9.8 Cap and shake the bottle to dissolve most of the NaCl. Not all of the NaCl will dissolve. A fine thin layer should remain at the bottom of the bottle.

9.9 Add the appropriate surrogate standard solution(s) (See Table 2) to all unknowns and quality control samples.

9.10 Add the appropriate LCS/MS/MSD spiking solution(s) (See Table 2) to the LCS, MS, and MSD.

9.11 Add the appropriate amount of solvent (See Table 1) to every sample and shake vigorously for a minimum of 45 seconds.

NOTE: The amount of extraction solvent may be altered to meet project specific objectives. If an alternate solvent volume is used, the surrogate and LCS/MS/MSD spiking volumes (See Table 1) should be proportionately adjusted. All sample, solvent, and spiking volumes must be documented in LIMS and the batch bench sheet.

9.12 Allow the sample to set for a minimum of 10 minutes and repeat the shaking again for a minimum of 45 seconds.

9.13 Allow sample to set for a minimum of 30 minutes. Then add DI water to bring the solvent up into the neck of the extraction vessel. Then let the sample set for another 10 minutes.

9.14 Transfer most of the solvent extract to a 20-mL scintillation vial or a 12 mL storage vial containing a small amount of Na_2SO_4. The solvent is not concentrated prior to sample analysis.

NOTE: Do not transfer any of the sample water to the scintillation vial.

NOTE: If an emulsion occurs, pull the emulsion up into a disposable pipet and squirt back into the bottle. Doing this action several times will usually break up the emulsion adequately to obtain solvent to inject. Also try breaking the emulsion mechanically by moving the disposable pipet back and forth through the emulsion.

NOTE: The use of sodium sulfate may be omitted if the sample extract is being treated with acid in accordance with PRE-004.

9.15 The sample is now ready to be cleaned up, if necessary, or analyzed by the determinative method.

10 QUALITY CONTROL

10.1 Any reagent blanks, matrix spikes, or replicate samples should be subjected to exactly the same analytical procedures as those used on actual samples.

10.2 An analytical batch consists of 20 or fewer samples. Quality control samples should be analyzed with each set with the following frequency:

Blanks - One per 20 or fewer samples, minimum one per day
LCSs - One per 20 or fewer samples, minimum one per day
MS/MSDs - One MS/MSD per 20 or fewer samples, minimum one set per day

Note: If an MS/MSD cannot be prepared because of limited sample volume, an LCS duplicate must be prepared.

10.3 Method blanks consist of an aliquot of laboratory reagent water processed through every step of the extraction process.

10.4 Laboratory control samples consist of an aliquot of laboratory reagent water spiked with the determinative method target compounds, prepared and processed through every step of the extraction process.

10.5 Matrix spike/matrix spike duplicate samples consist of duplicate aliquots of sample spiked with the determinative method target compounds, prepared and processed through every step of the process.

10.6 Surrogates are added to every sample and QC sample in the batch in accordance with the determinative method.

11 METHOD PERFORMANCE

11.1 Refer to the determinative methods for performance data.

12 WASTE MANAGEMENT / POLLUTION PREVENTION

12.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.

13 REFERENCES

13.1 Method 3511; Organic Compounds in Water by Micro-extraction, SW-846, Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA, OSWER; November 2002.

TABLE 1

SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative Method	Initial pH	Initial Vol. (mLs)	Salted[1]	Initial Solvent	Final Extract Vol.
8015 DRO	- [2]	400	Yes	Hexane	5
8081A	5-9	200	Yes	Iso-octane	5
8081A TCLP	NA	40	Yes	Iso-octane	10
8082	5-9	400	Yes	Iso-octane	5
8270D Alkylated PAHs	5-9	400	Yes	Hexane	10

[1] Amount of salt used is based on initial volume in mLs. See Section 9.6.
[2] The pH must be checked and documented, but pH adjustment is unnecessary.

TABLE 2

SURROGATE AND LCS/MS/MSD INFORMATION

Determinative Method	Surrogate Spike Concentration (µg/mL)	Surrogate Spike Amount (µL)	LCS/MS/MSD Spike Concentration (µg/mL)	LCS/MS/MSD Spike Amount (µL)
8015 DRO	To be defined	To be defined	To be defined	To be defined
8081A (standard list)	6.0	50	0.8/1.6/3.2/8.0	100
8081A (standard list TCLP)	6.0	100	0.8/1.6/3.2/8.0	200
8081A (Mix 2)	6.0	50	1/2	100
8081A (toxaphene)	6.0	50	100	100
8081A (toxaphene TCLP)	6.0	100	100	200
8081A (tech. chlordane)	6.0	50	40	50
8081A (tech. chlordane TCLP)	6.0	100	40	100
8082 (1242)	6.0	50	100	50
8082 (1248)	6.0	50	100	50
8082 (1254)	6.0	50	100	50
8082 (1260)	6.0	50	100	50
8270D Alkylated PAHs	5	500	10	50

The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede any prior revisions effective immediately as dated.

Reviewed By:

Karl Olm, Operations Manager — 03/21/16 Date

Patrick Letterer, Quality Manager — 03/21/16 Date

Approved By:

Nick Nigro, General Manager — 03/21/16 Date

P-23

Self-Contained Soil Extraction (Rev.0.4;03/28/2016; PRE-003)

METHOD 3570

SELF-CONTAINED SOIL EXTRACTION

1 SCOPE AND APPLICATION

1.1 This method describes a procedure for extracting organic compounds from soil and sediment samples.

1.2 This method is applicable to the extraction of organic compounds in preparation for a variety of chromatographic procedures.

1.3 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2 SUMMARY OF METHOD

2.1 Soil and sediment samples are dried with sodium sulfate and extracted with a solvent as listed in Table 1.

3 DEFINITIONS AND ACRONYMS

3.1 There are many terms and acronyms used throughout this document. Check the definitions and acronyms sections of the Quality Manual for complete explanations.

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in the extracted ion chromatograms. Reagent blanks are analyzed with every analysis batch to monitor for potential interferences with a goal of the procedure being interference free.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the ECCS Chemical Hygiene Plan (CHP), and this document. Refer to the CHP for more detailed safety information or for information not listed in this document.

5.2 Eye protection that protects against splash and appropriate gloves should be worn while samples, standards, solvents, and reagents are being handled during this procedure.

5.3 Employees must handle glassware and equipment carefully in order to prevent injury and accidents. Any damaged or broken glassware is to be discarded or moved to the glass repair box.

5.4 ECCS maintains a Material Safety Data Sheet (MSDS) for every chemical used in the laboratory. The MSDS file is kept in the main laboratory.

6 APPARATUS AND MATERIALS

6.1 Balances:

6.1.1 Top loader - capable of weighing to 0.01 g

6.2 Vials:

6.2.1 12 mL storage vials with Teflon® lined screw cap

6.2.2 20 mL glass scintillation vials

6.2.3 40 mL VOA vials

6.2.4 60 mL VOA vials

6.3 Syringes: Various sizes, 10-1000 µL, Gastight

6.4 Repeater Plus with Combitips of various sizes (Eppendorf or equivalent)

6.5 Glass disposable transfer pipettes - 5 3/4" and 9" and rubber bulbs

6.6 Assorted laboratory glassware

6.7 Stainless steel spatulas

6.8 Optifix solvent dispensers capable of dispensing accurate volumes (EM Science or equivalent)

6.9 Refrigerator capable of maintaining 4 °C

6.10 Freezer capable of maintaining temperatures below -15 °C

6.11 Muffle furnace capable of maintaining 400 ºC

7 REAGENTS

7.1 Reagent grade chemicals shall be used in all tests. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

7.2 Solvents

7.2.1 Dichloromethane, CH_2Cl_2 - pesticide quality or equivalent

7.2.2 Acetone, CH_3COCH_3 - pesticide quality or equivalent

7.2.3 Iso-octane, C_8H_{18} – pesticide quality or equivalent

7.2.4 Hexane, C_6H_{14} – pesticide quality or equivalent

7.2.5 Solvent mixes

7.2.5.1 80% Iso-octane/20% acetone: Combine 3200 mL of iso-octane with 800 mL of acetone in a 4 L solvent bottle. Document the preparation in LIMS, label appropriately, and assign a 1 year expiration date.

7.2.5.2 90% Dichloromethane/10% acetone: Combine 3600 mL of dichloromethane with 400 mL of acetone in a 4 L solvent bottle. Document the preparation in LIMS, label appropriately, and assign a 1 year expiration date.

7.3 Solid Reagents

7.3.1 Sodium sulfate (Na_2SO_4) – reagent grade

7.3.1.1 Sodium sulfate is baked in a muffle furnace at 400 °C for a minimum of 4 hours and is stored in sealed glass containers prior to use. Label each container with the LIMS ID.

7.3.2 Sand

7.3.2.1 Sand is baked in a muffle furnace at 400 °C for a minimum of 4 hours and is stored in sealed glass containers prior to use. Label each container with the LIMS ID.

7.4 Acids and Bases: Not applicable to this method

7.5 Surrogate spiking solution

7.5.1 See Table 1 for surrogate spiking information for each definitive method.

7.5.2 Refer to the definitive method for surrogate preparation instructions.

7.6 Laboratory control sample (LCS)/matrix spike/matrix spike duplicate (MS/MSD) spiking solution

7.6.1 See Table 1 for LCS and MS/MSD spiking information for each definitive method.

7.6.2 Refer to the definitive method for LCS and MS/MSD spiking solution preparation instructions.

8 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Soil samples should be collected in amber glass jars with Teflon-lined caps and refrigerated at 4 °C after collection. Soil samples may be collected in plastic Ziploc® bags for onsite analysis if regulatory rules allow.

8.2 Soil samples should be extracted within 14 days of collection. Soil samples for most compounds of interest can be frozen for longer periods until extraction if regulatory rules allow. Store extracts in a freezer at -15 °C or below and analyze within 40 days of extraction.

9 PROCEDURE

9.1 See Table 1 for specific definitive method information.

9.2 Remove the samples to be extracted, and the surrogate and matrix spiking solutions from cold storage and allow to come to room temperature.

9.3 Prepare a minimum of one blank, LCS, and MS/MSD with each set of 20 or fewer samples extracted per day. Use silica sand for the blank and LCS.

NOTE: If a single extraction is being performed for multiple definitive methods, prepare separate samples for the LCS, MS, and MSD per each definitive method.

9.4 Soil samples are typically not homogenous. The sample should be mixed as thoroughly as possible prior to weighing. Eliminate stones and other debris from samples.

9.4.1 Weigh 5.0 g of sample into a LIMS labeled and tared 20 mL scintillation vial. Add 5 or more grams of anhydrous sodium sulfate to dry the sample and mix well. Wet silt or clay soils may require over 10 g of sodium sulfate to dry the sample.

NOTE: A larger vial can be used if necessary.

9.4.2 Drying the sample completely to a free-flowing powder consistency with sodium sulfate is CRITICAL. A poorly dried sample will lead to poor recoveries of target analytes.

9.4.2.1 Occasionally a sample will not dry adequately in the 20 mL vial. Re-weigh the sample in a 40 mL or 60 mL VOA vial and mix with additional sodium sulfate to obtain a dry sample. If this procedure is used the extraction solvent volume will have to be increased resulting in increased limits of detection. The amount of surrogate added to this sample is also adjusted accordingly. Record the information on the batch bench sheet in LIMS.

NOTE: If the drying procedure isn't working effectively, cap the vial and allow it to sit on the bench anywhere from 30 minutes to overnight.

9.5 Add the appropriate surrogate standard solution(s) (See Table 1) to all unknowns and quality control samples.

NOTE: If a single extraction is being performed for multiple definitive methods, the surrogate(s) for each method must be added.

9.6 Add the appropriate LCS/MS/MSD spiking solution(s) (See Table 1) to the LCS, MS, and MSD samples.

NOTE: If a single extraction is being performed for multiple definitive methods, prepare separate samples for the LCS, MS, and MSD per each definitive method.

9.7 Check that the Optifix is set precisely by measuring with a 10 mL Class A volumetric flask. Record the check on the batch bench sheet in LIMS.

9.8 Add 10.0 mL of appropriate extraction solvent (See Table 1), shake for 45-60 seconds, and allow settling for 2 minutes. Shake again for 45-60 seconds.

9.9 Allow samples to settle for a minimum of 10 minutes. The samples are now ready for analysis.

NOTE: Sample extracts (i.e. solvent) may be transferred to LIMS labeled 12 mL amber storage vials, if desired.

9.10 Samples are stored frozen by LIMS extraction batch number in the sample extract freezer.

10 QUALITY CONTROL

10.1 Any reagent blanks, matrix spikes, or replicate samples should be subjected to exactly the same analytical procedures as those used on actual samples.

10.2 An analytical batch consists of 20 or fewer samples. Quality control samples should be analyzed with each set with the following frequency:

Blanks - One per 20 or fewer samples, minimum one per day
LCSs - One per 20 or fewer samples, minimum one per day
MS/MSDs - One MS/MSD per 20 or fewer samples, minimum one set per day

10.3 Method blanks consist of an aliquot of sand processed through every step of the extraction process.

10.4 Laboratory control samples consist of an aliquot of sand spiked with the determinative method target compounds, prepared and processed through every step of the extraction process.

10.5 Matrix spike/matrix spike duplicate samples consist of duplicate aliquots of sample spiked with the determinative method target compounds, prepared and processed through every step of the process.

10.6 Surrogates are added to every sample and QC sample in the batch in accordance with the determinative method.

11 METHOD PERFORMANCE

11.1 Refer to the determinative methods for performance data.

12 WASTE MANAGEMENT / POLLUTION PREVENTION

12.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.

13 REFERENCES

13.1 Method 3570; Micro-Scale Solvent Extraction (MSE), Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA, OSWER; November 2002.

TABLE 1

SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative Method	Surrogate Spike Concentration (µg/mL)	Surrogate Spike Amount (µL)	LCS/MS/MSD Spike Concentration (µg/mL)	LCS/MS/MSD Spike Amount (µL)	Extraction Solvent	Final Extract Vol.
8015 DRO		To be defined		To be defined	Hexane	10
8081A (standard list)	6.0	100	0.8/1.6/3.2/8.0	100	80/20 I/A	10
8081A (Mix 2)	6.0	100	1/2	100	80/20 I/A	10
8081A (toxaphene)	6.0	100	100	100	80/20 I/A	10
8081A (tech. chlordane)	6.0	100	40	50	80/20 I/A	10
8082 (1242)	6.0	100	100	100	80/20 I/A	10
8082 (1248)	6.0	100	100	100	80/20 I/A	10
8082 (1254)	6.0	100	100	100	80/20 I/A	10
8082 (1260)	6.0	100	100	100	80/20 I/A	10
8141A Pesticides Mix 1	20	100	4/10/20	500	80/20 I/A	10
8141A Pesticides Mix 2	20	100	2/4/10/20	500	80/20 I/A	10
8141A Pesticides Mix 3	20	100	2/4/10/20	500	80/20 I/A	10
8141A Organophosphates (FPD)	20	100	10/20/50	100	80/20 I/A	10
8270D Pesticides	10	100	20[1]	100[1]	80/20 I/A	10
8270D Alkylated PAHs	50	50	10	200	90/10 M/A	10
8270D Explosives	100	200	50	400	80/20 I/A	10
8270D Pyrethroids Mix 1	50	50	25	100	80/20 I/A	10
8270D Pyrethroids Mix 2	50	50	17.8/20/23.8	100	80/20 I/A	10
8095 Nitro-Aromatics (NPD)	400	100	100	100	80/20 I/A	10
8095 Nitro-Aromatics (ECD)	20	100	10	200	80/20 I/A	20

[1] For 8270D Pesticides, LCS/MS/MSD samples must be spiked with both Mix 1 and Mix 2 spiking solutions. The concentration of the spiking standards and the amount to be spiked is the same.

80/20 I/A = 80% iso-octane/20% acetone
90/10 M/A = 90% dichloromethane/10% acetone

The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede any prior revisions effective immediately as dated.

Reviewed By:

03/21/16

Karl Olm, Operations Manager — Date

03/21/16

Patrick Letterer, Quality Manager — Date

Approved By:

03/21/16

Nick Nigro, General Manager — Date

P-24

Determination of
Total Solids
(Rev.5.4; 02/02/2016;
GEN-007)

DETERMINATION OF TOTAL SOLIDS

1 PURPOSE

1.1 This standard operating procedure (SOP) establishes the procedure for performing a total solids analysis.

2 SCOPE AND APPLICATION

2.1 This SOP applies to the analysis of samples that require results to be reported on a dry weight basis. Most if not all soils, solids, and sediments received by ECCS require a total solids determination.

3 ACCOUNTABILITY

3.1 The Operations Manager shall be accountable for this SOP. All employees shall be responsible for implementation of the SOP.

4 SUMMARY OF METHOD

4.1 When a sample is being prepared for analyses of target constituents, a separate aliquot will be removed for the determination of dry weight. Approximately 15 to 30 grams of sample are placed in a pre-weighed drying pan and dried in a convection oven overnight or to a constant weight at 105 °C.

4.2 Samples known or suspected to contain significant concentrations of toxic, flammable or explosive material should not be oven dried because of safety concerns.

5 APPARATUS

5.1 Disposable aluminum pans

5.2 Drying oven capable of maintaining a temperature of 100 – 110 °C

5.3 Top loading balance capable of weighing to 0.01 g

5.4 Stainless steel spatulas

5.5 Thermometer

6 PROCEDURE

6.1 Use the LIMS to batch the samples that will be analyzed. See Figure 1 for an example of a LIMS generated bench sheet. If the LIMS is unavailable, use FORM GEN-007-1.

6.2 Record the sample number on the aluminum pan using a Sharpie.

6.3 Make sure the balance has been checked for calibration today. If not, check the calibration in accordance with the logbook.

6.4 Tare the balance, weigh the empty pan, and record the sample identification (ID) and weight on the LIMS generated Dry Weight Bench Sheet or on FORM GEN-007-1. Also complete the date, balance ID and analyst information required on the bench sheet.

6.5 Place approximately 15 to 30 grams of solid sample in the drying pan. Record the weight to the nearest 0.01 g.

NOTE: Samples known or suspected to contain significant concentrations of toxic, flammable, or explosive material should not be oven dried because of safety concerns. If a sample contains high levels of target constituents or solvents, consult the Operations Manager about how to proceed. Samples that have a safety risk can be reported on an "as is" basis or air dried overnight in the hood.

6.6 Record the initial oven temperature in the logbook once the oven has reached the operating range of 100° to 110° C.

6.7 Dry the sample at 105 °C overnight (minimum 8 hours) or to a constant weight if shorter than 8 hours.

NOTE: Constant weight requires repeating the drying cycle of drying, cooling, and weighing until a constant weight is obtained or until the weight change is less than 2% of the previous weight or 0.3 g, whichever is less.

6.8 Record the final oven temperature in the logbook when removing the samples.

6.9 Remove the pan(s) from the drying oven and allow cooling for 15-30 minutes or until the sample(s) reaches room temperature.

6.10 Weigh the dry sample and record the final weight on the bench sheet.

6.11 Enter the initial pan weight, initial pan + sample weight, and final pan + sample weight from the bench sheet into the LIMS for each sample.

6.12 Sign the bench sheet.

6.13 The final % solids result is calculated by the LIMS or with a calculator using FORM GEN-007-1 as follows:

$$\%\,Solids = \frac{g\ of\ dry\ sample}{g\ of\ wet\ sample} \times 100$$

7 QUALITY CONTROL

7.1 A duplicate sample should be prepared at a minimum of one per day or twenty samples whichever is more frequent.

7.1.1 The relative percent difference (RPD) between the sample and the duplicate must be determined. The calculation is as follows:

$$RPD = \frac{R1 - R2}{\frac{(R1 + R2)}{2}} \; X \; 100$$

Where: R1 = sample percent solids value
R2 = duplicate percent solids value

7.1.2 The RPD between the sample and the duplicate must be less than or equal to 20%. If the RPD is > 20%, the samples in the set must be repeated.

7.1.3 A sample that has percent solids > 100 must be reanalyzed.

8 REFERENCE

8.1 2540B, Total Solids Dried at 103-105 ^{o}C, Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

8.2 ASTM D2974-87, Standard Test Method for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, Annual Book of ASTM Standards, March/April 1993.

Figure 1: Example LIMS Generated Bench Sheet

DRY WEIGHT BENCH SHEET

A601025

ECCS

Printed: 1/31/2016 7:21:18PM

Matrix: Soil

Prepared using: WETCHEM-OLD - % Solids

Lab Number	Sample Name	Pan (g) [Response]	Pan + Wet Sample (g) [RT]	Pan + Dry Sample (g) [MDA]

Date/Time In: _____ Weighed By: _____ Balance ID: _____ Oven ID: _____

Date/Time Out: _____ Weighed By: _____ Balance ID: _____ Oven Temp. (C): _____

Page 1 of 1

The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

Reviewed By:

01/26/16

Karl Olm, Operations Manager — Date

01/26/16

Patrick Letterer, Quality Manager — Date

Approved by:

01/26/16

Nick Nigro, General Manager — Date

P-25

Determination of Total Suspended Solids
(Rev.3.6; 02/02/2016; LAM-012)

DETERMINATION OF TOTAL SUSPENDED SOLIDS (TSS)

1 SCOPE AND APPLICATION

1.1 The total suspended solids determination represents the amount of solid material in the sample minus the contribution of free water and other volatile material. This method is applicable to drinking, surface, and domestic and industrial wastewaters.

Reported Analyte	STORET Number
Total Suspended Solids	00530

1.2 The practical range of this determination is 4 mg/L to 20,000 mg/L. Actual reporting limits will be determined based on project requirements and sample volume used. A typical reporting limit is 40 mg/L.

1.3 The analyst should have completed training and demonstrated capability prior to analyzing samples. Training includes an understanding of this method and generating an acceptable PT study result.

2 SUMMARY OF METHOD

2.1 A 250 mL aliquot of sample is passed though a pre-weighed filter and dried in a convection oven overnight so that a constant weight is reached. The increase in weight of the filter represents the total suspended solids. Larger sample volumes can be used when necessary to meet client specific reporting limit needs.

2.2 Samples known or suspected to contain significant concentrations of toxic, flammable or explosive material may not be applicable to this method and should not be dried because of safety concerns. The analyst should use caution in this regard. The drying oven should be vented or located in a fume hood for proper ventilation to protect nearby workers from potential hazardous fumes.

3 DEFINITIONS AND ACRONYMS

3.1 There are many terms and acronyms used throughout this document. Check the definitions and acronyms sections of the Quality Manual for complete explanations.

4 INTERFERENCES

4.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.

4.2 Samples high in dissolved solids (filterable residue), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting and cleaning the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.2.6) minimizes this interference.

4.3 Exclude large floating particles or submerged conglomerates of non-homogeneous materials if it is determined that their inclusion is not representative.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the ECCS Chemical Hygiene Plan (CHP), and this document. Refer to the CHP for more detailed safety information or for information not listed in this document.

5.2 Eye protection that protects against splash and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled during this procedure. Lab coats are recommended.

5.3 Employees must handle glassware and equipment carefully in order to prevent injury and accidents. Any damaged or broken glassware is to be discarded or moved to the glass repair box.

5.4 ECCS maintains a Material Safety Data Sheet (MSDS) for every chemical used in the laboratory. The MSDS file is kept in the main laboratory.

6 APPARATUS AND MATERIALS

6.1 Disposable aluminum drying pans, VWR part # 25433-008, or equivalent

6.2 Drying oven capable of maintaining 105 °C

6.3 Analytical balance, capable of weighing to 0.1 mg or better

6.4 42.5 mm glass fiber filter disks, without organic binders, Whatman (1827-042), or equivalent.

6.5 Thermometer

6.6 Desiccator

6.7 Filtering apparatus with reservoir and coarse (40-60 µm) fritted disk as a filter support

6.8 Tweezers

6.9 250 mL graduated cylinder

6.10 Vacuum pump

6.11 Squirt bottle

7 REAGENTS AND STANDARDS

7.1 Distilled or de-ionized water is used for pre-rinsing the filters, for blanks, and for rinsing the apparatus.

8 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Non-representative particulates such as leaves, sticks, and large biological organisms should be excluded from the sample if it is determined that their inclusion is not desired in the final result.

8.2 Preservation of the sample is not practical; the holding time is seven days, but analysis should begin as soon as possible.

8.3 Samples should be refrigerated to 4 °C to minimize microbiological decomposition of solids.

9 PROCEDURE

9.1 Preparation of glass fiber filters:

9.1.1 Place two filters on the suction bed and attach the reservoir.

9.1.2 Turn on the vacuum to approximately five inches of Hg and rinse the filter with three 50 mL aliquots of DI water.

9.1.3 Increase the vacuum to approximately 15 inches of Hg for one minute to remove most of the water.

9.1.4 Release the vacuum slowly until vented and detach the reservoir portion of the filtering apparatus.

9.1.5 Transfer the two filters to a labeled aluminum drying pan using tweezers. Place the pan containing the filters in a clean pie tin or other clean metal pan for transport to and from the oven. Repeat the process with additional filters and pans as needed.

9.1.6 Heat the drying oven to 103-105°C. Place the pans/filters into the drying oven overnight, or a minimum of eight hours. If the filters are dried less than eight hours it must be demonstrated that they have come to constant weight by two successive weightings a minimum of one hour apart.

9.1.7 From this step and forward always use a tweezers to move the aluminum pans/filters.

9.1.8 Remove the pan/filters from the oven and place in a desiccator near the analytical balance to cool.

9.1.9 Using a calibrated four place analytical balance, weigh each numbered tin containing the glass microfiber filters to 0.0001 g and record the weight on the TSS log page. Always use a tweezers for grasping and moving the aluminum drying pan.

9.1.10 If the filters were not dried for a minimum of eight hours, place the pan/filters back into the oven for a minimum of one hour. Repeat the weighing until a constant weight is obtained (weight loss is less than 0.0005 g). Record the weights on the TSS log page. Enter the final weight, in mg, into LIMS under "RT".

9.2 Sample vacuum filtration:

9.2.1 Transfer the glass microfiber filters from the aluminum drying pan to the suction base using a tweezers. Verify that the glass microfiber filters are centered over the fritted disk filter support portion of the suction base.

9.2.2 Position and attach the reservoir portion of the filtering apparatus over the glass microfiber filters and suction base.

9.2.3 Position and attach the vacuum apparatus to the filtering apparatus and apply the vacuum.

Note: Vacuum should be run at less than five inches of Hg.

9.2.4 Wet the glass microfiber filters with a small volume of distilled water to seat them against the fritted disk filter support.

9.2.5 Shake the aqueous sample vigorously and transfer 250 mL of sample with a graduated cylinder to the reservoir portion of the filtering apparatus. The aqueous sample is filtered through the glass microfiber filter disks. Rinse the graduated cylinder with three 10 mL volumes of DI water and add each to the reservoir. Record the volume of sample filtered on the TSS log page. Enter the volume used, in mL, into LIMS under "Response".

NOTE: A larger volume of sample may be required if the captured residue is less that 1.0 mg, or if lower reporting limits are needed. Sample volume provided by the client may limit how much is available for total suspended solids analysis.

9.2.6 With the vacuum on, rinse the reservoir funnel wall with three 10 mL volumes of distilled water allowing complete drainage.

9.2.7 Increase the vacuum to approx. 15 inches of Hg for approximately one minute to remove most of the water.

9.2.8 Release the vacuum slowly until vented and detach the reservoir portion of the filtering apparatus.

9.2.9 Carefully remove filters from the fritted disk filter support using a tweezers and place them in the labeled aluminum drying pan.

9.3 Sample drying:

9.3.1 Using a tweezers, place the pans/filters into the drying oven overnight, or a minimum of eight hours. If the filters are dried less than eight hours it must be demonstrated that they have come to constant weight by two successive weighings a minimum of one hour apart.

9.3.2 Using a tweezers, remove the pan/filters from the drying oven and place them in a desiccator to cool.

9.3.3 Using a tweezers, place the pan containing the filters on the analytical balance and record the weight on the TSS log page.

9.3.4 If the filters were not dried for a minimum of eight hours, using a tweezers place the pan/filters back into the oven for a minimum of one hour. Repeat weighings until a constant weight is obtained (weight loss is less than 0.0005 g). Record the weights on the TSS log page. Enter the final weight, in mg, into LIMS under "MDA".

9.4 Calculate the total suspended solids result as follows:

$$Total\ Suspended\ Solids\ (mg/L) = \frac{W_f - W_i}{V} x1000\ mL/L$$

Where: W_i = Initial constant weight of pan and filters (mg)
W_f = Final constant weight of pan, residue and filters (mg)
V = Volume of sample filtered (mL)

10 QUALITY CONTROL

10.1 A duplicate sample must be prepared at a minimum of one per day or twenty samples whichever is more frequent, assuming adequate sample volume is available. Percent RPD should be within 10%.

$$RPD = \frac{R1 - R2}{\left(\frac{R1 + R2}{2}\right)} \times 100$$

Where: R1 = Sample result
R2 = Duplicate result

10.2 A method blank using 250 mL of DI water should be prepared at a minimum of one per day or twenty samples whichever is more frequent. The acceptance criterion is less than the reporting limit.

11 METHOD PERFORMANCE

11.1 Acceptable performance testing (PT) sample results have been generated and are on file in the laboratory.

11.2 Precision data are not available at this time.

12 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

12.1 Contingencies for out-of-control data should be evaluated on a case-by-case basis. A Corrective Action Form (CAF) must be completed for those times that acceptable QC results cannot be achieved. The CAF must be completed by the analyst and filed with the Quality Manager. Analytical results shall be qualified as necessary.

13 WASTE MANAGEMENT / POLLUTION PREVENTION

13.1 All waste will be disposed of in accordance with federal, state, and local regulations. This method has been prepared to minimize the waste produced and the potential for pollution of the environment. All ECCS employees shall follow this method and the guidance provided in the ECCS Health and Safety manual.

14 REFERENCES

14.1 2540 D. Total Suspended Solids Dried at 103-105°C, Standards Methods for the Examination of Water and Wastewater, 20th Edition, pp 2-57 – 2-58, 1998.

14.2 Code of Federal Regulations, Pt. 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants, (7-1-03 Edition).

Pace Analytical®
ECCS Mobile Lab Services

TOTAL SUSPENDED SOLIDS BENCH SHEET

Sample ID	Sample Volume (V) in mL	Pan and Filters Initial Weight (W_i) in g[a]	Pan and Filters Initial Weight (W_i) in g[a] (if needed)	Pan and Filters Initial Weight (W_i) in g[a] (if needed)	Pan, Residue and Filters Final Weight (W_f) in g[a]	Pan, Residue and Filters Final Weight (W_f) in g[a] (if needed)	Pan, Residue and Filters Final Weight (W_f) in g[a] (if needed)
Weighing Number		1	2	3	4	5	6
LIMS Column	Response	RT	RT	RT	MDA	MDA	MDA
Blank							

Weighing Number	**Temperature In**	**Date and Time In**	**Temperature Out**	**Date and Time Out**	**Balance ID**	**Weighed By**
1						
2						
3						
4						
5						

TOTAL SUSPENDED SOLIDS BENCH SHEET

6						

[a] Record weights in LIMS in mg ex (1.5395g = 1539.5mg)

The signatures below indicate the following individuals have reviewed this document in its entirety and authorize its use to supersede prior revisions as of the effective date of this SOP.

Reviewed By:

01/26/16

Karl Olm, Inorganics Team Leader, Operations Manager — Date

01/26/16

Patrick Letterer, Quality Manager — Date

Approved By:

01/26/16

Nick Nigro, General Manager — Date

E-01

Environmental Sample Receipt and Unpacking (Rev. 17; 11/02/2018; S-SA-WI10725)

eurofins	Always check on-line for validity. **Environmental Sample Receipt and Unpacking**	Level: **Work Instruction**
Document number: **S-SA-WI10725**		
Old Reference: **1-P-QM-PRO-9015504; DOD - SOP-SA-103**		
Version: **17**		Organisation level: **5-Sub-BU**
Approved by: **UCSS** Effective Date **02-NOV-2018**	Document users: **5_EUUSLA_Sample Administration_Manager, 6_EUUSLA_ Sample Administration_Sample Unpacking**	Responsible: **5_EUUSLA_Sample Administration_Manager**

Revision Log
Cross Reference
Purpose
Scope
Personnel Training and Qualification
Procedure

Revision Log

Revision:	17	Effective Date:	This version
Section	Justification		Changes
Revision Log	Formatting requirement		Removed revision logs up to the previous version
Procedure	Reflect current practices		Replaced references to SA (Sample Administration) with SR (Sample Registration)
Procedure A	Reflect current practices		Clarified who needs to write the returned time on the driver pickup list
Procedure B	Reflect current practices		Added reference to verifying that each container is properly identified before removing from bags.
Procedure C	Reflect current practices		Added reference use of numbered table markers on carts
Procedure D	Reflect current practices		Clarified how client services was notified of problems with incoming samples
Procedure F	Reflect current practices		Changed "bad smell" to "strong odor"
Procedure G	Reflect current practices		Added additional information on discarding damaged or contaminated coolers

Revision:	16	Effective Date:	Mar 15, 2018
Section	Justification		Changes
Revision Log	Formatting requirement		Removed revision logs up to the previous version
Procedure F	Reflect current practices		Added reference to availability of thermal and cut-resistant gloves

Cross Reference

Document	Document Title

Document	Document Title
M-FRM12743	Shipping Request
S-SA-WI10743	Taking the Temperature of Environmental Samples Upon Arrival at the Lab
QA-SOP11893	Environmental Hazardous Sample Communication Procedure

Purpose

Receipt of samples and documentation of receipt are very critical steps in the overall processing of samples. It is very important that proper handling procedures are established and closely followed to ensure the integrity of the sample is maintained and clients' needs are met. It is the responsibility of the Sample Receiving personnel to properly unpack, document, and communicate information about the samples to the Sample Registration (SR) personnel. All steps must be documented and traceable.

Scope

This SOP is designed to cover all procedures involved in proper receipt of and unpacking of environmental samples in SR.

Personnel Training and Qualification

All SR entry and sample receipt personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP, S-SA-WI10743 and QA-SOP11893.

Procedure

A. Documentation of receipt

All samples received via courier (i.e., FedEx, UPS, U.S. Mail, laboratory courier, etc.) must be documented on the Sample Administration Receipt Documentation Log (Doc Log). The Doc Log is generated through the Doc Log program in our LIMS system. All information must be accurate, clear, and complete.

Samples received via ELLE's (Eurofins Lancaster Laboratories Environmental) Transportation (samples picked up by ELLE's couriers) are also documented on the Transportation Pickup schedules. As each lab courier returns, the receipt person and the courier must verbally and visually verify that each sample group is accounted for as listed on their pickup list. The courier must write the time that they returned to the lab at the top of his/her corresponding sheets.

The chain of custody (COC) accompanying all samples must be signed in the “Received for ELLE by” space by the receipt person, or the last “Received by” portion of the chain.

B. Unpacking procedures

Begin by looking for the expected RUSH/SHORT HOLD samples for that day. Unpack those first using the procedures outlined below.

First, initiate the Doc Log for the environmental samples to be unpacked. A separate form must be generated for each submittal group of samples. A unique Doc Log ID number is generated as each new doc log is initiated. This ID is entered into the LIMS system during the sample entry process to associate each entry group with its corresponding doc log. The unpacker completes the form by identifying the client/project name, date of receipt, time of receipt, delivery method, number of packages, and state of origin. The next section of the Doc Log is the “Arrival Condition Summary” where the following conditions are documented with a “Yes” or “No” answer: whether the shipping container was sealed, whether a custody seal was present , whether the seal was intact, whether the package was chilled , whether paperwork was enclosed, whether samples were intact, whether any samples are missing, whether any extra samples are present, whether there are discrepancies in container quantity and type of container from the COC, whether sample ID’s on COC match containers, whether sample date/times match COC, whether, VOA vial headspace is present, and the ID’s of vials with headspace. The presence of any trip blanks with the submittal group is recorded, along with the type of trip blank. And lastly, there are some questions regarding whether air samples were received, and if so were any flow controllers received, quantity, and if there were any returned canisters and the corresponding ID numbers of those canisters. Samples received in our kits, coolers, or client packaging are opened and visually checked for shipping damage. If damage is encountered, follow procedures outlined in the Unpacking problems/paperwork discrepancy, Section D. Once visual assessment is complete, remove all packaging material and place it on the unpacking table.

Initially, remove all large sample containers from the cooler and place them on a cart or on the table, leaving all the 40 ml volatile vial containers in the cooler with the ice. Prior to unwrapping or removing sample containers from bubble wrap or baggies, verify that the individual containers are labeled with the sample identification and that the identification was not on the outside of the packaging only. If the identification is only on the outside of the packaging, keep the containers in the bag, do not remove them. Next, read the client COC and/or other accompanying paperwork and verify that all contents have been received, reconciling the larger containers first. If you encounter missing sample(s), broken samples, or sample/paperwork discrepancies, follow the Unpacking problems/paperwork discrepancy procedures, Section D. Once the larger containers have been reconciled against the client paperwork, remove the 40 ml volatile vials from the cooler and reconcile those containers. Lastly, remove all wet ice and ice packs from shipping container. The wet ice must be placed in the ice disposal tank. If client

paperwork is not included, retain some portion of the packaging for identification (i.e., shipping label, FedEx slips, etc.)

For samples received by ELLE's drivers, begin by scanning the drivers' pickup schedule for indications of fecal coliforms (6−hour holding time), RUSH, microbiological, or other short holding time analysis samples. Unpack those samples first. To quickly retrieve those or any other samples, look for the cooler number indicated on the pickup schedules and go to the corresponding cooler.

If the driver indicated that paperwork was included, make sure that you have that paperwork. Check samples received vs. the client paperwork. Again, if a discrepancy exists, follow the Unpacking problems/paperwork discrepancy procedures. If a discrepancy is found, check with the driver to see if he/she has any information or can help resolve the current problem or any similar problems in the future. Initial the driver schedule next to the "unpacker" space if, and only if, you actually unpacked that group of samples. At the end of the day photocopy all the drivers' schedules and return the originals to the Transportation supervisor.

Once the samples have been removed from the shipping containers and matched against the client paperwork (with any discrepancies properly documented), the unpacker places his/her signature in the final *received by* section of any client−submitted COC with the date and time of receipt at the laboratory. Lastly, the unpacker's name and their employee number will be recorded at the bottom of the Arrival Condition Summary section of the Doc Log, followed by the date and actual time of sample unpacking. The unpacker will print the Doc Log once all the information pertaining to that submittal group has been recorded and give the Doc Log and client's paperwork to the entry staff.

C. Placement of samples onto carts for entry

All groups are to be placed on carts. Multiple groups may be placed on the same cart as long as the groups are kept distinctly separate from other clients' sample groups. All samples are lined up according to the sample order on the client paperwork, or if none exists, in the most obvious manner. All glass vials must be put in foam holders and placed with the other bottles from the same sampling point. Place one of the numbered table markers with the group on the cart and write the corresponding marker number on the back of the client's COC. Print the doc log and take the client paperwork along with the doc log to the group leaders in the sample entry room. When each cart is full, or 30 minutes since samples were first placed on carts, or all samples are unpacked (whichever would come first), deliver the cart of samples to the SR personnel. If for some reason all SR personnel are unavailable, the carts of samples are placed within storage at the appropriate temperature conditions similar to how the samples were received (i.e., refrigerated, frozen, or room temperature).

D. Unpacking problems/paperwork discrepancies

In the case of a missing sample(s), the receipt person unpacking the samples must notify a SR person immediately. The SR person must also verify that the sample(s) are missing by completely checking the shipping container, the packaging contents, and comparing the paperwork and the received containers. Once the SR person and the unpacker are satisfied that the sample(s) are missing, the unpacker must begin documentation in the Doc Log program. By answering “Yes” to whether samples were missing, the “Details Editor” section of the program will open where the specific missing sample ID’s and comments can be recorded.

Broken samples must be removed from the shipping container and disposed of appropriately. In some cases the sample may be salvageable (i.e., a soil in a cracked container can be transferred to a new jar, etc.). The unpacker must judge this situation and if in question check with a SR person. Any information on completely broken sample containers or partially broken/transferred containers must be documented on the Doc Log. By answering “No” to whether samples are intact, the “Details Editor” section of the Doc Log will open where the specific sample IDs and comments can be recorded. Broken or severely damaged shipments may be photographed with a camera, if it is felt that we need to document sample condition. This is an optional piece of documentation, not required in all circumstances.

Sample vs. paperwork identification, container quantity, and/or date/time discrepancies must also be documented in the “Details Editor” section of the doc log. Any specific sample IDs and information on the containers which differs from the COC will be recorded.

The SR person must immediately communicate all problems that will affect the analysis (e.g. high temperature, mislabeling of containers, incorrect or wrong bottle, flipped collection times, missing samples, missing coolers, broken samples, and/or discrepancy problems) to the appropriate Client Services Representative (CSR) via email or phone call. Copies of the Doc Log and any client paperwork must be given to the CSR. Client Services must call the client and document the resolution. All Doc Logs are filed with the first page of the client acknowledgment and client paperwork.

E. Temperature of coolers

For all environmental samples, the temperature upon receipt is required documentation. See S-SA-WI10743 (Taking the Temperature of Environmental Samples Upon Arrival at the Lab).

F. Safety procedures

The following safety procedures must always be followed during the sample receipt process:

1. Latex or nitrile gloves must always be worn and replaced if torn or soiled.

2. Thermal gloves are available for use when handling samples received in dry ice. In addition, cut-resistant gloves are available for use when handling loose ice containing broken glass.

3. Lab coats and safety glasses must always be worn.

4. If a sample meets any of the following conditions, it must be placed in the hood:

   a. Strong odor

   b. Expanded, bulging containers or septums

   c. Fuels (neat)

   d. Labeled as hazardous

   e. Breakage and/or leakage

   (If ever in doubt, place in the hood until a technical decision can be made.)

5. Where warranted appropriate warning stickers must be used (i.e., Hazardous, Quarantined Soil). Guidelines for identifying and communicating information about potentially hazardous samples are in *QA-SOP11893*.

G. Cleaning of coolers and kits

1. ELLE coolers and kits

All coolers/kits must be completely cleaned after all the samples are unpacked.

a. All packaging material must be discarded properly.

b. All shipping tape and labels removed from the inside and outside of the cooler/kit, with the exception of the ELLE identification tag.

c. The cooler/kit must be completely wiped out so there is no remaining water in the cooler/kit.

d. The interior of the cooler/kit must be wiped down with a disinfectant to remove any residual moisture and visible debris so the cooler is reasonably clean and ready for future use.

e. The cooler/kits must be returned to the bottles department for future use.

f. In the event that a cooler/kit is damaged or contaminated it must be discarded and not used any further. To do so, place the cooler in the area outside of the unpacking room designated for "Coolers for the Dumpster"

g. Coolers that need to be discarded are taken to a designated spot out in the hallway where facility services will dispose of them.

2. Client coolers and kits

All client coolers/kits must be completely cleaned following the procedures above with the following exceptions:

a. Any temperature blanks, ice packs, and/or special packaging material supplied by the client will be returned in their cooler to them if they supplied a return shipping label or have supplied the lab with their courier account number.

b. Form *M-FRM12743* must be completed for return to the client with the client's commercial courier billing account number filled in on the form.

c. The coolers/kits are taken to the Shipping Department with the Shipping form taped to the lid of the corresponding cooler/kit.

M-FRM12743 Shipping Request
QA-SOP11893 Environmental Hazardous Sample Communication Procedure
S-SA-WI10743 Taking the Temperature of Environmental Samples Upon Arrival at the Lab

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End of document

Version history

Version	Approval	Revision information
15	21.MAR.2016	
16	01.MAR.2018	
17	30.OCT.2018	

E-02

Automated Storage, Retrieval, and Discarding of Samples (Rev. 10; 03/15/2017; S-SS-WI12042)

Revision log
Reference
Purpose
Scope
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Procedure

Revision log

10	**Effective Date:**	This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Procedure B.3.	Clarification	Added location of samples with Cart ID
Procedure B.7.	Enhancement	Added the Missing Samples-Requisition Contacts email information

Revision: 09	**Effective Date:**	Apr 22, 2011
Section	**Justification**	**Changes**
Revision Log	Formatting requirements per LOM-SOP-LAB-201	Removed revision logs up to the previous version.
Safety Precautions and Waste Handling	Formatting requirements per LOM-SOP-LAB-201	Added required text
Personnel Training and Qualification	Formatting requirements per LOM-SOP-LAB-201	Added required text

Reference

Chemical Hygiene Plan, Lancaster Laboratories, current version.

Purpose

The purpose of this SOP is to define correct procedures for the storage, retrieval and discard of samples using the Automated Storage and Retrieval System (ASRS) or the manual Sample Storage program.

Scope

This SOP covers the steps required to put samples in location, pull samples for analysis, and locate samples not found in the ASRS. It also outlines the procedures used for handling samples designated as “Hold at Discard” and removing samples upon their discard date for disposal.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal and state laws and regulations.

Information concerning the known toxicity, properties, or any special handling precautions can be found in the material safety data sheets (MSDS) available in Sample Storage or by a Safety Officer. The MSDS should accompany the samples when pulled for the technical departments. Safety glasses and sample storage lab coats are required as personal protective wear.

Hearing protection devices (earplugs) are available. However, occupational noise measurements indicate that the sound pressure level is below the Occupational Safety and Health Administration’s (OSHA) Action Level of 85 dB (A). The Action Level is the threshold above which OSHA mandates hearing protection.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP.

The initial training consists of observing all of the steps in this procedure carried out by an experienced ASRS operator. Next, the trainee performs all of the same steps while the experienced person watches, answers questions, and gives feedback. Following the initial training, an experienced operator is available as a resource until no longer required. Operators are considered proficient when all of the steps in the procedure can be carried out independently.

Procedure

A. Daily ASRS operations/pulling requisitions:

1. Log onto the system by typing your employee login and password.

2. Choose the *ASRS* option in the Function Panel.

3. Choose the *Pick* option in the ASRS Functions.

4. Requisitions are required to be batched before they can be processed; this queues the orders to the designated workstation to be pulled.

Choose the *Shipping* option on the Function Selection Panel and then select *Order Wave Release*, the rule that best describes the orders you wish to pull, and change the Order Status to *Batche*d.

5. To release orders, select the *GO* option on the Pick screen. This automatically pulls the orders that are currently batched. A Tote ID is printed as each new order is processed. Place the Tote ID with the order so the samples can be scanned to this ID.

6. As the containers are brought out to the workstation, scan the container ID to bring the grid up on the screen. All of the samples to fill the order are displayed in green; scan the Sample Bar Code and the Tote ID to remove the sample from the container and link it to the designated Tote ID.

NOTE: Discards can also be done at this time. See Procedure Section E.

7. When an order is finished, missing samples are displayed on a Discrepancy List found in Parallax. Any other queued orders are also processed.

8. Completed orders are stored in the appropriate queuing area until a lab technician comes to retrieve them.

B. Locating samples not in the ASRS

1. When all of the samples are retrieved from the ASRS, check the Discrepancy List to view any samples that are still needed to complete the order.

2. Samples previously assigned to another Tote ID are located on a cart waiting to be put back into the ASRS, or have not yet been returned from the lab.

3. Samples with a Cart ID are scanned to a fixed location and can be located in the back walk-in.

4. If no prior destination is given, the samples are new and should be located in the SA walk-in waiting to be labeled or processed by one of the other sample support areas. If the samples were already processed by one of the other areas, the samples should be on a put−away cart waiting to be put back into the ASRS.

NOTE: Samples that require a pH check, volatile prep, or homogenization must be communicated to that area prior to the samples being given to the technical areas.

5. Samples that are not located by the requisition time need to be communicated to the appropriate lab technician. Keep the technical department up−to−date on the status of missing samples. If the technician informs you that a sample is *Rush* status, the sample becomes a priority. If you require further assistance to find the sample, contact your supervisor.

6. If the sample is not located by the end of a shift, the ASRS operator is required to let the next shift know the status of the sample. If the sample is missing on your next shift, you must contact your supervisor.

7. If a sample is not found 24 hours prior to the deadline, the client service representative must be contacted to inform the client. These samples are indicated by the labs using the Missing Samples-Requisition Contacts email address which alerts this group that the sample must be located or a reason must be provided as to why the sample was not found.

C. Retrieving samples designated as client hold

1. Processing the Client Hold Orders

a. Log on to the system by typing your employee login and password.

b. Choose the *ASRS* option in the Function Panel

c. Choose the *Pick* option in the ASRS Functions

d. Client Hold requisitions are required to be batched before they can be processed; this queues the orders to the designated workstation to be pulled. This is done by choosing the

Shipping option on the Function Selection Panel. Then select *Order Wave Release*, chose the *Client Hold* orders, and change the status of the orders you wish to pull to *Released*.

NOTE: For Client Hold orders the *CLH* option must be selected as the conveyance type.

e. When orders are released, the *GO* option is selected on the Pick screen. This automatically pulls the orders that are currently batched. A Tote ID is printed as each new order is processed. The Tote ID is placed with the order and the samples are scanned to this ID. If multiple orders for the same client are placed in the same tote all Tote IDs must be displayed.

f. As the containers are brought out to the workstation, scan the container ID to bring the grid up on the screen. All of the samples needed to fill the order are displayed in green. Scanning the Sample Bar Code and the Tote ID removes the sample from the container and links it to the designated Tote ID. If a container is filled before the order is complete you can request a new Tote ID by scanning the next sample, using the mouse and right clicking on the proper order on the Put Bar and select *New Tote ID*.

NOTE: Discards can also be done at this time. See Section E.

g. When the order is finished, the box containing the samples must be taken over to the bulk workstation. There the samples are imported into the Client Hold area by inducting the Tote into an available location.

2. Scanning Totes in to the Client Hold Pod

a. Using the RF gun log in to the system.

b. On the Main Menu choose the *Ship* option.

c. Next choose *Matrix Induct.*

d. Scan the Tote you wish to put away and scan an Empty shelf location in the Client Hold Pod. You will get a question to Consolidate “Yes or No”. Always answer “Yes”.

e. If multiple Tote IDs are on the current box, scan each ID to the same location. The last ID scanned contains all the samples in the box. All previous IDs scanned must be crossed off with a sharpie so that only the last ID scanned is displayed.

NOTE: If a container is scanned to a location that is not full and has the same client and account number as a Tote you are currently scanning away, you can combine by:

f. Repeating steps 1 – 3 above.

g. Scan the Tote you wish to combine to the location in which the container resides.

h. All the samples from the existing container are transferred to the container ID that was recently scanned to the location; all samples need to be moved to the current box.

3. Discarding from containers in the Client Hold Pod

a. Remove the container from the designated location and write the shelf location on the container ID (this is to allow for the container to be returned to the proper location if all the samples aren't up for discard at this time).

b. Using the Evolution workstation, choose the *Bulk* option on the Function Selection Panel.

c. Navigate to Execute and Deplenish Container.

d. Choose the Existing Container option.

e. Scan the container ID.

f. Scan each sample located in the container. There are three scenarios that could take place:

(1) Sample is obsolete - remove from the container as normal and discard.

(2) Sample is obsolete and hazardous - remove from container and place on a designated hazardous cart.

(3) Sample has not reached the necessary time to be marked as obsolete and remains a client hold. In this case the system prompts you to scan a container. The options are:

(a) Scan the existing container. All samples designated as client hold remain in the current container.

(b) Scan an existing container that is scanned to a different Client Hold Location. All samples designated as Client Hold move to this container (samples must be same client and account number).

(c) With either option the container scanned remains unless a new container to discard from is selected.

g. Return the container to the designated shelf location if samples remain. If no samples remain the container ID must be removed and the box is not returned to the Client Hold Pod.

D. Sample put away

1. Log on to the system by typing your employee login and password.

2. Choose the *ASRS* option in the Function Panel.

3. Select the *Put Away* option in the ASRS Functions.

4. Choose *Put Away*, which displays the available empty locations for each cell configuration listed in order of container height.

5. Choose the container size you need and type the number of samples and press ENTER. This brings you the available containers for the size requested.

6. When the containers arrive, scan the container ID. The grid display on screen shows the available open locations.

7. Scan the Sample ID and the Open Location ID to code the sample away.

NOTE: Procedure D. Step 4 can be performed in any of the three main screens (Pick, Deplenish, and Put Away).

E. Sample discard procedure

NOTE: Discards can be processed through Parallax. In the SA function, choose *Download ASRS Discard File*. This starts a count of the samples that are up for discard and sends the file to *Evolution*.

1. Log on to the system by typing your employee login and password.

2. Choose the *ASRS* option in the Function Panel.

3. Select the *Deplenish* option in the ASRS Functions.

4. Select the *Discard* option which displays the available discards for each cell configuration listed in order of container height.

5. Choose the container size you wish to discard from and type the number of samples you need and press ENTER. This brings you the available containers for the size requested.

6. When the containers arrive, scan the container ID and the grid displays on the screen. The regular obsolete samples are displayed with blue lines going through the sample; hazardous are in red.

7. Scan the designated samples and separate upon removal from the ASRS and take to the appropriate area for disposal.

NOTE: Procedure E. Step 4 can be performed in any of the three main screens (Pick, Deplenish, and Put Away).

F. Disposing non-hazardous samples

1. Use discretion when disposing all non−hazardous samples. Do NOT discard a solid or liquid sample if it has an “off odor” or unusual appearance.

2. Liquid samples should essentially be clear and odorless like water. It is acceptable for the sample to have some discoloration and/or sediment present. Unless the sample has a solvent odor, an oily or viscous appearance, a paint odor or appearance, a lot of solids, etc., it may be poured into the discard sink. The ventilation hood and the cold water tap must be turned on. After pouring the sample out, place the container in the trash hopper.

3. Discard solid, non−hazardous samples into the trash hopper. Do not dump solid samples out of their containers. Discard containers unopened, with the contents intact, directly into the hopper.

4. If you have any doubt on the proper disposal of a sample, place it on the cart designated for questionable discard and notify you supervisor or the EHS (Environmental Health and Safety) Group for assistance.

G. Disposing of hazardous samples

1. Take the hazardous discard cart to the Sample Storage workstation.

2. Log on to Parallax.

3. Using the SA function on the tool bar, choose the *Sample Storage* option.

4. When in this program, choose *Hazardous Discard Assessment*.

5. Scan the barcode on one of the hazardous samples.

6. If the sample discard date has not come up yet, a window appears indicating “Sample has not been discarded.” If applicable, the hazardous results are displayed. Before disposing, further investigation must be done to see if the sample can be discarded.

7. If the sample does **not** have any Final Results that exceed the adjusted Hazardous Limit for that particular analysis, a window appears indicating **“No hazardous analysis in this sample.”** Read the lab notes very carefully at this point because the sample may be hazardous for reasons other than the analyses performed.

8. If the sample has no hazardous analyses or lab note indicating hazard, check the matrix of the sample and the sample designation to be sure that the sample matrix is not something that must not be dumped down the sink (i.e., oils, paint, or paint thinner, ink, etc.).

9. If the sample is flagged as *Hazardous* and the discard date has passed, the following information appears on the screen:

Master Analysis Number
Piece Analysis Number
Analysis Name
Final Result
Reporting Units
Discard Date
Adjusted Hazardous Limit

10. Based on the data provided from the Assessment Program, the sample needs to be put into one of the following waste streams:

Waste Stream	Description
Acid Wastes	pH <4; no heavy metals; dumped in acid tank
Solvent Wastes	A liquid flagged as hazardous due to an analysis result regulated under TCLP Waste Characteristic List (except heavy metals); dumped in solvent waste drum
Labpack Wastes	A solid or liquid flagged as hazardous that does not fit into one of the other categories
Hazardous & Quarantine Soil Wastes	A solid (soil) flagged as hazardous due to heavy metals or solvents; NO PCBs
Waters containing Heavy Metals (free liquid)	Liquid flagged as hazardous due to Heavy Metals (Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, or Silver); liquid poured off into a drum
Waters containing Heavy Metals (in vials)	Liquid samples in vials flagged as hazardous due to Heavy Metals (Arsenic, Barium, Cadmium, Chromium, Lead, Mercury, Selenium, or Silver)
Waters containing Cyanides	Sample vials flagged for cyanide-containing compounds
PCB-containing Soils	Solid samples (and containers) flagged for PCBs
PCB-containing Water & Oils	Liquid samples (and containers) flagged for PCBs
Soil Extracts in Methanol	Place vials in open-head drum labeled: “GC/HPLC and 40−mL vials containing organic solvents”
Soil Extracts in Sodium Bisulfate	Place vials in open-head drum labeled: “Soil extracts containing Sodium Bisulfate”

11. If you have any doubt on the proper waste stream for disposal of a sample, place it on the cart designated for questionable hazardous discard and notify your supervisor or the EHS (Environmental Health and Safety) Group for assistance.

12. Lancaster Laboratories reserves the right to return samples to the submitting client if they contain high levels of hazardous substances or do not fit into one of our existing waste streams.

End of document

Version history

Version	Approval	Revision information
9	22.APR.2011	
10	01.MAR.2017	

E-03

Laboratory Sample Analysis Record (LSAR) Documentation
(Rev. 8; 05/04/2018; QA-SOP11184)

eurofins	Always check on-line for validity.	Level:
Document number: **QA-SOP11184**	**Laboratory Sample Analysis Record (LSAR) Documentation**	**Standard Operating Procedure**
Old Reference: **1-P-QM-QMA-9017318; LOM-SOP-ES-212**		
Version: **8**		Organisation level: **5-Sub-BU**
Approved by: **UDM6** Effective Date **04-JUN-2018**	Document users: **4_EUUSLA_ELLE_All_Technical, 5_EUUSLA_Client Services_All, 5_EUUSLA_Sample Bottles_All, 6_EUUSLA_ Client Services_Project Management**	Responsible: **5_EUUSLA_Env Quality Assurance_All**

Revision Log
Reference
Cross Reference
Purpose
Scope
Definitions
Personnel Training and Qualifications
Procedure

Revision Log

Revision: 8		**Effective Date:**	This version
Section	**Justification**		**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356		Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in D4		Replaced all EtQ references with D4 references and added hyperlinks
Table of Contents	No longer used		Table of Contents is not required

Revision: 7		**Effective Date:**	Jul 30, 2013
Section	**Justification**		**Changes**
Revision Log	Formatting requirements per LOM-SOP-LAB-201		Removed revision logs up to the previous version.
Throughout Document	Reflect re-identification of documents in EtQ		Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Throughout Document	Company name change		Remove and/or change text citing “Lancaster Laboratories, Inc.” or “LLI”

Reference

Environmental Quality Policy Manual, current version.

Cross Reference

Document	Document Title
S-SA-FRM10722	Environmental Sample Administration Receipt Documentation Log
QA-SOP11914	Legal Chain-of-Custody Documentation
G-FAC-SOP12733	Building Security

Purpose

There are regulatory and client requirements to ensure that samples are not tampered with while in the lab’s possession. To address these requirements, the laboratory uses employee

pass key building security measures, electronic sample requisition records, and documentation of the processes performed on a sample. The Laboratory Sample Analysis Record documentation is provided to clients to show dates, times, and personnel involved in the preparation and analysis of samples.

Scope

This procedure applies to the processing/transfer of laboratory samples. It details the laboratory's responsibilities relative to external COC documentation and the generation and reporting of the Laboratory Sample Analysis Record (LSAR) document. Legal (or forensic) Chain of Custody (COC) documentation is addressed in *QA-SOP11914*. The LSAR is provided with every Analysis Report.

Definitions

1. Analysis Request/Environmental Services Chain of Custody: Document used to track submission of the sampling containers to the client and receipt of samples from the client to the laboratory. This may also be referred to as an External COC.

2. Custody: A sample is in custody if it is in any one of the following states:

   a. In actual physical possession

   b. In view after being in physical possession

   c. Locked up so no one can tamper with it

   d. In a secured area, restricted to authorized personnel (e.g., in the ASRS)

3. Laboratory Sample Analysis Record (LSAR): Documentation of the date, time, and analyst for each sample preparation and analysis. The information is compiled in the LIMS using electronic records tracking from the data upload and entry functions. This displays, per sample, on each Analysis Report.

4. Legal Chain of Custody (COC): See *QA-SOP11914*. This additional level of documentation may be required for litigation or forensic work.

Personnel Training and Qualifications

All personnel responsible for sample processing must read this SOP. Documentation of the reading must be made in the employee of departmental training files.

Procedure

A. External COC documentation

1. This documentation begins with the preparation of sampling containers. The person packing the bottle order for shipment to the client initiates the External COC documentation. If the delivery of containers is via the laboratory's Transportation Department, the Sample Container Record (SCR) Number (written on Section 6 of the External COC) is utilized to track the person preparing the bottle order. The laboratory drivers must sign Section 9 when they relinquish the bottles to the client. Drivers must also sign the forms when they pick up samples from the client for transportation to the laboratory.

2. When samples arrive at the laboratory for analysis, a member of the Sample Administration (SA) department receives them and signs the external COC form that accompanies the samples, if provided. If our Transportation department picked up the samples, the driver must sign the COC to relinquish the samples to Sample Administration.

3. The Sample Administration staff tracks the custody of samples between receipt and entry into LIMS on Form *S-SA-FRM10722*. The client's sample designation is used for identification purposes until a unique laboratory number is assigned.

4. Samples are entered into the LIMS as described in *Environmental Quality Policy Manual*. Sample Administration will enter an analysis number for "Legal Chain of Custody" if requested. A lab note will inform analysts of the need for Legal COC documentation. This note will also be automatically printed on the sample labels.

5. After the original copy of the external client COC/analysis request form is scanned into the LIMS, it is filed with the client paperwork. If requested, the original copy of the external client COC/analysis request form will be sent to the client with their report.

B. Building security

1. Building access is restricted to employees or visitors that have signed in through security and are escorted during their time in the building. See *G-FAC-SOP12733* for details.

2. Each employee is given an identification card which is coded with defined levels of building access. The main sample storage locations have restricted access to authorized employees only.

C. Sample identification and traceability

1. All samples are labeled with a unique 7 digit identification number and a bar code that identifies the tests to be performed and the main storage location.

2. The sample number is used in all preparation and analysis steps and will link the date, time, and analyst name to each test on the raw data, instrument records, and in the LIMS.

D. Laboratory sample analysis record (LSAR)

1. As part of data entry into the LIMS, the analyst name and the dates and times for any preparation and analyses are stored electronically for each sample. This information may be part of the upload file from the instrument to the LIMS or it may be manually entered by the analyst.

2. This record provides documentation of the processes performed on each sample, when it occurred, and the responsible person.

3. The information is compiled in the LIMS through the report generation function and is documented per sample on each client's Analysis Report.

G-FAC-SOP12733 Building Security
QA-SOP11914 Legal Chain-of-Custody Documentation
S-SA-FRM10722 Environmental Sample Administration Receipt Documentation Log

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End of document

Version history

Version	Approval	Revision information
7	30.JUL.2013	
8	21.MAY.2018	

E-04

Management of Hazardous Wastes in the Laboratory (Rev. 4; 09/04/2018; G-EHS-SOP22000)

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Revision Log

Revision: 4	**Effective Date:**	This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Procedure A. and C.	New RCRA 2017 regulation	Updated all affected labeling requirements for waste containers
Procedure F.1.	Additional buildings added to campus and correct location verbiage	Updated all waste storage locations with Building Id information
Procedure F.8.	Reflect current process	Removed logging of waste amounts as this process has been obsoleted with updating billing
Procedure G.	Re-classify emergencies	Clarified updated policy on requirements of spill/emergencies requiring a 1-1-1 call

Revision: 3	**Effective Date:**	Nov 03, 2015
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects re-identification of documents in EtQ Reflects applicable company name	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers Replaced *Lancaster Laboratories* with *Eurofins Lancaster Laboratories*
Procedure A.5.	Clarification	Added EHS to be consulted for container choice
Procedure D.3.	Clarification	Added dates should not be added until the container is full and ready for removal
Procedure E.2.	Clarification	Added all hazardous waste labels must be removed or defaced before disposal
Procedure F.1.	Reflect current process	Updated responsibilities
Procedure F.3.	Reflect current process	Updates made to the container types used
Procedure F.3.p.	Reflect current process	Updated EHS will perform as necessary for all departments
Procedure F.3.	Reflect current process	Updated responsibilities in NOTE
Procedure F.4.	Reflect current process	Updated to include authorized personnel and with adequate lighting
Procedure F.6.	Reflect current process	Replaced “Rubber” with “TyChem” apron type
Procedure F.6.c.	Reflect current process	Added nitrile gloves
Procedure F.7.	Clarification	Added number to call
Procedure F.8.	Clarification	Added “if applicable (GC vial)”

Reference

1. USEPA, Resource Conservation and Recovery Act (RCRA), 40 CFR Parts 260-272.

2. Commonwealth of Pennsylvania, Hazardous Waste Regulations, PA Code, Title 25, Environmental Resources, Hazardous Waste Regulations, July 1980, Chapters 260-270.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
1-P-QM-QMA-9017344	Environmental, Health, and Safety (EHS) Assessments
1-P-QM-QMA-9017347	Incident Response Plan

Purpose

Ensure hazardous wastes are handled and disposed of in accordance with Federal and State regulations and to prevent the risk of exposure and/or injury to employees and the environment.

Scope

This standard operating procedure applies to the accumulation of hazardous wastes in the laboratory and their transfer from the laboratory to the hazardous waste storage areas.

Definitions

1. Acutely Hazardous Waste – A RCRA listed waste that is regulated due to its level of hazard to people and/or the environment (see Table I).

2. Hazardous Waste (HW) – Regulated under RCRA as a listed waste or having hazardous characteristics: ignitibility, reactivity, corrosivity, and toxicity.

3. PADEP – Commonwealth of Pennsylvania Department of Environmental Protection; the department which performs compliance and enforcement of the USEPA regulations.

4. USEPA – United States Environmental Protection Agency; the Federal agency which establishes environmental policies and regulations.

5. Satellite Accumulation Container – A RCRA approved container at or near a hazardous waste generating process. The container must be under the control of the operator of that process.

6. Satellite Accumulation Area (SAA) – An area within the laboratory facility where hazardous wastes are temporarily stored. Laboratory areas physically separated from one another (i.e., by walls and doors) are considered individual accumulation areas. More than one type of waste may be accumulated per area. The maximum, total volume allowed per SAA is 55 gallons of hazardous waste(s) OR one quart of acute hazardous waste (see Table I).

NOTE: This includes wastes to be reclaimed or recycled.

7. Hazardous Waste Storage Area – Location(s) where compatible hazardous wastes are stored prior to being shipped off-site for treatment, storage, or disposal. The maximum duration that hazardous wastes may be stored in a HW Storage Area is 90 calendar days. These areas are locked and access is limited to employees with RCRA Hazardous Waste Transfer training.

Personnel Training and Qualifications

Employees who accumulate hazardous waste in the laboratory and/or transfer hazardous waste from the satellite accumulation areas to the hazardous waste storage areas must read this SOP. Additionally, completion of *Chemical Hygiene Plan* Training is required prior to handling any hazardous waste and annual RCRA Hazardous Waste Transfer Training is required for any employee who needs access to a storage area.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations. See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

1. At a minimum, standard laboratory protection (safety glasses, lab coat, and gloves) must be worn when handling hazardous wastes. Use of additional or higher levels of protective equipment (face shield, plastic apron, etc.) is recommended when pouring large volumes of liquid wastes.

2. You must never dump any waste into a hazardous waste container unless you know the wastes are compatible. If uncertain, ask a supervisor or EHS

before making a decision to dump waste into a hazardous waste storage container.

Procedure

A. Container Management

1. Never completely fill any container with wastes. Always leave adequate headspace for gas expansion.

2. Never store a waste container in direct sunlight.

3. It is extremely important to keep incompatible wastes segregated.

4. Waste containers must be maintained in good condition (i.e., no rust, dents, cracks, leaks, etc.).

5. Each department is responsible for purchasing an adequate number of waste collection containers that are resistant to corrosion, leakage, or spills. Container construction must be compatible with the hazardous wastes they contain. Because of their high durability and chemical resistance, high-density polyethylene disposal cans are highly recommended for the disposal of acids and solvents. Prior to purchase, EHS should be consulted to approve container choice to ensure usability for waste disposal.

6. All waste containers must be clearly labeled with hazard classification labels (GHS, RCRA or DOT) and a black hazardous waste label indicating the container contents (i.e., nitric acid, miscellaneous inorganic acid waste, miscellaneous organic solvents, flammable GC vials, broken thermometers, etc.).

7. Empty reagent containers may be reused for hazardous waste collection provided they have been cleaned and dried and the original reagent label has been removed or completely covered.

8. All hazardous waste containers, including satellite accumulation containers, must be closed except when wastes are added or removed.

B. Secondary Containment

1. Containers used for instrument effluent must have secondary containment.

A polyethylene bucket capable of containing the entire volume of the collection container is acceptable.

2. Although not required in all cases, secondary containment for all satellite accumulation containers is considered a prudent practice and is strongly encouraged.

C. Labeling Requirements

1. All satellite accumulation containers must be labeled with hazard classification labels (GHS, RCRA or DOT) and a “Satellite Accumulation” black hazardous waste label indicating the container contents (i.e., nitric acid, miscellaneous inorganic acid waste, miscellaneous organic solvents, flammable GC vials, broken thermometers, etc.). Labels are available through the Waste Team.

2. The labels must be affixed to the outside of the container immediately upon being put into service.

3. The “Satellite Accumulation” label must contain a specific description of the container contents.

4. Placement of the labels should be away from the spout or mouth of the container to prevent smearing or damage of the label when pouring.

5. Where secondary containment is used and the outside of the waste container is not visible, all labels must be affixed to the outside of the secondary containment to mirror the satellite waste can.

D. Accumulation Time and Quantity Limitations

1. Labs generating multiple waste streams may have more than one satellite accumulation container. While there is no limit to the number of different satellite accumulation containers per area, the absolute minimum is strongly encouraged.

2. No more than 55-gallons of hazardous waste or one-quart of acutely hazardous waste (see table I for a list of Acutely Hazardous Wastes) may be stored in a satellite accumulation area regardless of the number of different waste streams generated or number of containers in the accumulation area.

3. Once 55 gallons of hazardous or one quart acutely hazardous waste is exceeded in the accumulation area, the full container or excess waste must be moved from the satellite accumulation area to a hazardous waste storage area within 72 hours.

Any satellite accumulation container moved to a final storage area must have the date added to the satellite accumulation label or the satellite accumulation label must be replaced with a completed “Hazardous Waste Accumulation” label (white and black lettering on a white background and red border. Dates should not be added until the container is full and ready for removal by EHS waste team.

E. Inspection and Documentation

1. All satellite accumulation containers shall be inspected each time waste is added to the SAA container for evidence of leaks, corrosions, dents, bulges, or other signs of loss of integrity and proper labeling. A formal inspection must be performed on a monthly basis, at a minimum, during the department monthly self -audit in accordance with 1-P-QM-QMA-9017344.

2. Any container with evidence of loss of integrity may not be reused and must be discarded. All hazardous waste labels must first be removed or defaced before disposing of containers in trash.

3. For labs accumulating multiple waste streams, each satellite accumulation container in the satellite accumulation area must be inspected.

4. Conduct an inventory to ensure that the 55-gallon hazardous or one-quart acute volume limit has not been exceeded.

5. In the event of a leak or spill, notify the EHS Office or the Emergency Response Coordinator immediately by dialing 1-1-1 from any facility phone.

6. Any missing or illegible hazardous waste accumulation labels must be replaced immediately.

F. Waste transfer

1. The EHS waste team is responsible for transferring hazardous wastes from the laboratory to the appropriate hazardous waste storage area. Each department may designate additional personnel to perform waste transfer as needed. EHS will provide RCRA training and access to the hazardous waste storage areas for Waste Team staff and Emergency Response Personnel.

Hazardous Waste Storage areas include Building L, Building I (Doors 1 & 3), USDA permitted quarantine soils Waste Storage in Building H, Building E, Building F, Building J, Building U and the South Campus Medical Waste Storage Building.

2. The hazardous waste storage area(s) shall be locked at all times, except when wastes are actively being transferred. Access privileges to the hazardous waste storage areas are granted upon successful completion of RCRA Waste Transfer training. Annual refresher training is required to maintain access privileges.

3. Eurofins Lancaster Laboratories has the following segregated hazardous waste streams. The type of storage container is indicated with each type of waste:

a. Flammable and toxic solvents (55-gallon conductive HDPE drums)

b. Acid Waste (elemental neutralization system)

c. Mercury salts in sulfuric acid / COD analysis waste (30-gallon HDPE closed top drum)

d. Hazardous and quarantine soils (55- gallon fiber drums)

e. Flammable GC/HPLC vials, no PCBs (55-gallon fiber drums)

f. Flammable GC/HPLC vials containing PCBs (55-gallon fiber drums)

g. Solvent spill clean-up (30-gallon oily waste cans)

h. Acid spill clean-up (30-gallon oily waste cans)

i. Corrosive organic extract vials (55-gallon HDPE drums)

j. Water containing heavy metals (30-gallon HDPE drums)

k. PCB-containing soils, oils, and waters (storage container varies depending on volume)

l. Vials containing cyanides (storage container varies depending on volume)

m. Chemically contaminated sharps (Fiberboard boxes)

n. Dioxin samples (storage container varies depending on volume)

o. GC/HPLC vials with Dioxins (storage container varies depending on volume)

p. Miscellaneous lab pack wastes including, but not limited to: expired chemicals, elemental mercury, mercury-containing wastes, and any other chemical-containing wastes which do not fit into one of the waste streams designated above (bottles, bags, buckets, etc.).

NOTE: The transfer of miscellaneous lab pack wastes from the facility to the waste storage will be performed by the EHS waste team as necessary from all departments.

4. Transfer of wastes to the waste storage areas located outside of the building must be performed by authorized personnel and with adequate lighting.

5. During the transfer of wastes to the centralized waste storage area, the following personal protective equipment (PPE) shall be worn:

a. Safety glasses

b. Lab coat

c. Latex or nitrile gloves

6. The following additional PPE is recommended for the transfer of acid and solvent waste into the final waste container. This PPE is available in the waste containment area. If this PPE is not available, contact the EHS Office immediately.

a. Face shield

b. TyChem apron

c. Butyl or nitrile or neoprene gloves

7. Care shall be exercised throughout the transfer process to avoid splashing or spilling of the waste. Spills must be cleaned up completely and immediately. Report all spills to the EHS Office or call 111 immediately.

8. Employees must inspect the area to verify all containers are closed and there are no leaks or spills. The centralized waste storage area shall be locked

upon leaving the room. Employees should contact the EHS Department with any questions or concerns.

9. All wastes should be properly identified and profiled. Contact EHS with any questions regarding new or modified waste streams to complete a waste profile questionnaire.

G. Emergencies Involving Hazardous Waste

1. Any accident or unusual incident involving hazardous waste shall be reported to the emergency response coordinator (ERC) or the Environmental, Health, and Safety (EHS) office. To report an emergency, dial 111 from any phone on the campus. Provide the following details to the emergency responder that answers the call: your name, a brief description of the incident, the location of the incident, information regarding any injured personnel and a phone number where you can be reached. The necessary Emergency Response Team personnel will be dispatched to your location.

2. If the problem is minor (e.g., a bench top spill or a spill of limited quantity in containment) and has been cleaned up, the person or persons involved in the incident may make the report verbally or in writing to the ERC or the EHS Office as soon as possible. If the problem is more serious or assistance is needed for remedial action, the ERC shall be contacted immediately.

3. In the event of a major spill (on the floor, out of containment OR on a person), do not attempt to clean up the spill unless you received advanced spill response training. Call 111 and the ERC will dispatch the laboratory spill response team. The spill response team has the specialized skills and equipment necessary to clean up hazardous spills.

4. In the event of a fire, pull the nearest fire alarm and dial 911 or 111. Small fires may be extinguished by using a fire extinguisher; however, fire extinguishers are not kept in the solvent sheds or waste containment areas because of the potential for explosion once an ignition source is present. In case of a fire in one of these locations, evacuate the area immediately and call 911 or 111.

5. If the emergency threatens human life or the environment, activate the community Emergency Management System (EMS) by dialing 911. The emergency operator will dispatch the County HAZMAT Team and the local Fire Company. Any calls to EMS should be reported immediately to the ERC and the president of Eurofins Lancaster Laboratories. Any spill of greater than 5 gallons of any toxic material or 10 gallons of any ignitable material must be reported to the

Department of Environmental Protection Emergency Hotline at 877-333-1904. These contacts will be the responsibility of the EHS Office or the president of the company.

6. Upon receiving notification of a serious accident, the ERC shall inform the president or other members of executive management in accordance with the Incident Response Plan, 1-P-QM-QMA-9017347.

Attachment:
Table I

End of document

Version history

Version	Approval	Revision information
4	04.SEP.2018	

Acetaldehyde, chloro-
Acetamide, N-(aminothioxomethyl)-
Acetamide, 2-fluoro-
Acetic acid, fluoro-, sodium salt
1-Acetyl-2-thiourea
Acrolein
Aldicarb
Aldicarb sulfone
Aldrin
Allyl alcohol
Aluminum phosphide
5-(Aminomethyl)-3-isoxazolol
4-Aminopyridine
Ammonium picrate
Ammonium vanadate
Argentate(1-), bis(cyano-C)-, potassium
Arsenic acid H_3AsO_4
Arsenic oxide As_2O_3
Arsenic oxide As_2O_5
Arsenic pentoxide
Arsenic trioxide
Arsine, diethyl-
Arsonous dichloride, phenyl-
Aziridine
Aziridine, 2-methyl-
Barium cyanide
Benzenamine, 4-chloro-
Benzenamine, 4-nitro-
Benzene, (chloromethyl)-
1,2-Benzenediol, 4-[1-hydroxy-2-(methylamino) ethyl]-
Benzeneethanamine, alpha,alpha-dimethyl-
Benzenethiol
7-Benzofuranol, 2,3-dihydro-2,2-dimethyl-, methylcarbamate
Benzoic acid, 2-hydroxy-, compd. with (3aS-cis)-1,2,3,3a,8,8a -hexahydro-1,3a,8-trimethylpyrrolo [2,3-b]indol-5-yl methylcarbamate ester (1:1)
2H-1-Benzopyran-2-one, 4-hydroxy-3-(3-oxo-1-phenylbutyl)-, & salts, when present at concentrations greater than 0.3%
Benzyl chloride
Beryllium powder
Bromoacetone
Brucine
2-Butanone, 3,3-dimethyl-1- (methylthio)-, O-[methylamino)carbonyl] oxime
Calcium cyanide
Calcium cyanide $Ca(CN)_2$
Carbamic acid, [(dibutylamino)-thio]methyl-, 2,3-dihydro-2,2-dimethyl- 7-benzofuranyl ester
Carbamic acid, dimethyl-, 1-[(dimethylamino) carbonyl]- 5-methyl-1H- pyrazol-3-yl ester
Carbamic acid, dimethyl-, 3-methyl-1-(1-methylethyl)-1H- pyrazol-5-yl ester.

Carbonic dichloride
Carbosulfan.
Chloroacetaldehyde
p-Chloroaniline
1-(o-Chlorophenyl)thiourea
3-Chloropropionitrile
Copper cyanide
Copper cyanide Cu(CN)
m-Cumenyl methylcarbamate
Cyanides (soluble cyanide salts), not otherwise specified
Cyanogen
Cyanogen chloride
Cyanogen chloride (CN)Cl
2-Cyclohexyl-4,6-dinitrophenol
Dichloromethyl ether
Dichlorophenylarsine
Dieldrin
Diethylarsine
Diethyl-p-nitrophenyl phosphate
O,O-Diethyl O-pyrazinylphosphorothioate
Diisopropylfluorophosphate (DFP)
1,4,5,8-Dimethanonaphthalene,1,2,3,4,10,10-hexa- chloro-1,4,4a,5,8,8a,-hexahydro-,(1alpha,4alpha,4abeta,5alpha,8alpha,8abeta)-
1,4,5,8-Dimethanonaphthalene,1,2,3,4,10,10-hexa- chloro-1,4,4a,5,8,8a-hexahydro-,(1alpha,4alpha,4abeta,5beta,8beta,8abeta)-
2,7:3,6-Dimethanonaphth[2,3-b]oxirene, 3,4,5,6,9,9-hexachloro- 1a,2,2a,3,6,6a,7,7a-octahydro-, (1aalpha,2beta,2aalpha,3beta, 6beta, 6aalpha,7beta, 7aalpha)-
2,7:3,6-Dimethanonaphth [2,3-b]oxirene, 3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-, (1aalpha,2beta,2abeta,3alpha, 6alpha, 6abeta,7beta, 7aalpha)-, & metabolites
Dimethoate
alpha,alpha-Dimethylphenethylamine
Dimetilan.
4,6-Dinitro-o-cresol, & salts
2,4-Dinitrophenol
Dinoseb
Diphosphoramide, octamethyl-
Diphosphoric acid, tetraethyl ester
Disulfoton
Dithiobiuret
1,3-Dithiolane-2-carboxaldehyde, 2,4-dimethyl-, O- [(methylamino)-carbonyl] oxime.
Endosulfan
Endothall
Endrin
Endrin, & metabolites
Epinephrine
Ethanedinitrile
Ethanimidothioc acid, 2-(dimethylamino)-N-

Ethyl cyanide
Ethyleneimine
Famphur
Fluorine
Fluoroacetamide
Fluoroacetic acid, sodium salt
Formetanate hydrochloride.
Formparanate
Fulminic acid, mercury(2+) salt
Heptachlor
Hexaethyl tetraphosphate
Hydrazinecarbothioamide
Hydrazine, methyl-
Hydrocyanic acid
Hydrogen cyanide
Hydrogen phosphide
Isodrin
Isolan
3-Isopropylphenyl N-methylcarbamate.
3(2H)-Isoxazolone, 5-(aminomethyl)-
Manganese,bis(dimethylcarbamodithioato-S,S')-,
Manganese dimethyldithiocarbamate
Mercury, (acetato-O)phenyl-
Mercury fulminate
Methanamine, N-methyl-N-nitroso-
Methane, isocyanato-
Methane, oxybis[chloro-
Methane, tetranitro-
Methanethiol, trichloro-
Methanimidamide, N,N-dimethyl-N'-[3-[[(methylamino)-carbonyl]oxy]phenyl]-, monohydrochloride
Methanimidamide, N,N-dimethyl-N'-[2-methyl-4-[[(methylamino)carbonyl]oxy]phenyl]-
6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro- 1,5,5a,6,9,9a-hexahydro-,3-oxide
4,7-Methano-1H-indene, 1,4,5,6,7,8,8-heptachloro -3a,4,7,7a-tetrahydro-
Methiocarb.
Methomyl
Methyl hydrazine
Methyl isocyanate
2-Methyllactonitrile
Methyl parathion
Metolcarb.
Mexacarbate.
alpha-Naphthylthiourea
Nickel carbonyl
Nickel carbonyl $Ni(CO)_4$, (T-4)-
Nickel cyanide
Nickel cynaide $Ni(CN)_2$
Nicotine, & salts
Nitric oxide
p-Nitroaniline

Nitroglycerine
N-Nitrosodimethylamine
N-Nitrosomethylvinylamine
Octamethylpyrophosphoramide
Osmium oxide OsO_4, (T-4)-
Osmium tetroxide
7-Oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid
Oxamyl
Parathion
Phenol, 2-cyclohexyl-4,6-dinitro-
Phenol, 2,4-dinitro-
Phenol, 2-methyl-4,6-dinitro-, & salts
Phenol, 2-(1-methylpropyl)-4,6-dinitro-
Phenol, 2,4,6-trinitro-, ammonium salt
Phenol, 4-(dimethylamino)-3,5-dimethyl-, methylcarbamate (ester).
Phenol, (3,5-dimethyl-4-(methylthio)-,methylcarbamate
Phenol, 3-(1-methylethyl)-, methylcarbamate.
Phenol, 3-methyl-5-(1-methylethyl)-,methyl carbamate.
Phenylmercury acetate
Phenylthiourea
Phorate
Phosgene
Phosphine
Phosphoric acid, diethyl 4-nitrophenylester
Phosphorodithioic acid, O,O-diethylS-[2-(ethylthio)ethyl] ester
Phosphorodithioic acid, O,O-diethylS-[(ethylthio)methyl] ester
Phosphorodithioic acid, O,O-dimethyl S-[2-(methylamino)-2-oxoethyl] ester
Phosphorofluoridic acid, bis(1-methylethyl) ester
Phosphorothioic acid, O,O-diethyl O-(4-nitrophenyl) ester
Phosphorothioic acid, O,O-diethyl O-pyrazinyl ester
Phosphorothioic acid,O-[4-[(dimethylamino) sulfonyl]phenyl] O,O-dimethyl ester
Phosphorothioic acid, O,O,-dimethyl O-(4-nitrophenyl) ester
Physostigmine
Physostigmine salicylate
Plumbane, tetraethyl-
Potassium cyanide
Potassium cyanide K(CN)
Potassium silver cyanide
Promecarb
Propanal, 2-methyl-2-(methylthio)-, O-[(methylamino)carbonyl] oxime
Propanal, 2-methyl-2-(methyl-sulfonyl)-, O-[(methylamino)carbonyl] oxime
Propanenitrile
Propanenitrile, 3-chloro-

Propargyl alcohol
2-Propenal
2-Propen-1-ol
1,2-Propylenimine
2-Propyn-1-ol
4-Pyridinamine
Pyridine, 3-(1-methyl-2-pyrrolidinyl)-, (S)-, & salts
Pyrrolo[2,3-b]indol-5-ol,1,2,3,3a,8,8a-hexahydro-1,3a,8-trimethyl, methylcarbamate (ester), (3aS-cis)-
Selenious acid, dithallium(1+) salt
Selenourea
Silver cyanide
Silver cyanide Ag(CN)
Sodium azide
Sodium cyanide
Sodium cyanide Na(CN)
Strychnidin-10-one, & salts
Strychnidin-10-one, 2,3-dimethoxy-
Strychnine, & salts
Sulfuric acid, dithallium(1+) salt
Tetraethyldithiopyrophosphate
Tetraethyl lead
Tetraethyl pyrophosphate
Tetranitromethane
Tetraphosphoric acid, hexaethyl ester
Thallic oxide
Thallium oxide Tl_2O_3
Thallium(I) selenite
Thallium(I) sulfate
Thiodiphosphoric acid, tetraethylester
Thiofanox
Thioimidodicarbonic diamide $[(H_2N)C(S)]_2NH$
Thiophenol
Thiosemicarbazide
Thiourea, (2-chlorophenyl)-
Thiourea, 1-naphthalenyl-
Thiourea, phenyl-
Tirpate.
Toxaphene
Trichloromethanethiol
Vanadic acid, ammonium salt
Vanadium oxide V_2O_5
Vanadium pentoxide
Vinylamine, N-methyl-N-nitroso-
Warfarin, & salts, when present at concentrations greater than 0.3%
Zinc, bis(dimethylcarbamodithioato-S,S')-
Zinc cyanide
Zinc cyanide $Zn(CN)_2$
Zinc phosphide Zn_3P_2, when present at concentrations greater than 10%
Ziram

E-05

Microwave Extraction Method 3546 for PCBs in a Solid Matrix
(Rev. 8; 05/01/2018; T-OE-PEST-WI10927)

eurofins	Always check on-line for validity. **Microwave Extraction Method 3546 for PCBs in a Solid Matrix**	Level: **Work Instruction**
Document number: **T-OE-PEST-WI10927**		
Old Reference: **1-P-QM-WI -9015104**		
Version: **8**		Organisation level: **5-Sub-BU**
Approved by: **UKL3** Effective Date **01-MAY-2018**	Document users: **5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Pest/PCB Soils, 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID

Analysis - 10497, 11140, 13100

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Revision Log

Revision 8		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Purpose	Removed section	Not required
Scope	Reflects current practice	Added tissues to matrix types
Apparatus and equipment	Reflects current practice	Removed graduated cylinder. Added glass vial inserts

Revision 8		**Effective Date:** This version
Throughout Procedure	Clarification	Updated wording to clarify steps
Procedure 1	Reflects current practice	Added weights for tissue matrix and clarified the range
Procedure 15	Reflects current practice	Combined the copper and florisil steps as they are done together now.
Procedure 16	Reflects current practice	Updated the bottling requirements

Revision: 7	**Effective Date:**	**03-DEC-2015**
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Historical/Local Document Number	Added new analysis to procedure	Add 13100 to historical document number.
Procedure	Updated to current process	Added information about sample aliquot

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3546, Revision 0, February 2007.

2. *Chemical Hygiene Plan*, current version.

3. MARS Operation Manual, Revision 2, February 2006.

Cross Reference

Document	Document Title
Analysis #*0042, 1030, 6011, 7512, 10225, 10736, 10906, 12800, 13236*	Polychlorinated Biphenyls (PCBs) by Method 8082 in Solids and Wipes
Analysis #*2487*	Food and Tissue Preparation
T-OE-PEST-WI11410	Pesticide Extract Cleanup Using GPC by method 3640A
T-OE-GEN-WI10864	Glassware Cleaning for Organic Extractions
T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-OE-GEN-WI10876	Organic Extraction Standards Storage and Handling

Scope
This procedure is applicable for the extraction of PCBs from soils, tissues, or solid wastes.

Basic Principles
A portion of sample to be analyzed is placed in an extraction vessel. Surrogate standards are added to each sample to monitor recovery. The vessel is then loaded into the microwave and extracted. The organic compounds present in the sample dissolve in the solvent, which is then removed. The sample is then concentrated and bottled.

Several cleanup procedures may be required to eliminate matrix interferences before the sample can be analyzed. They include: sulfuric acid treatment, florisil, copper, and gel-permeation cleanup (GPC).

Reference Modifications
The KD is not removed from the steam bath and cooled before adding hexane. The Snyder column is not removed for the solvent addition; therefore, the KD does not have to be cool.

Double volumes of surrogates and matrix spiking solutions are not added when a sample requires gel-permeation cleanup. Instead, the extract is concentrated to one-half the normal final volume after GPC to make up for the loss on GPC and maintain the limit of quantitation.

The Joint of the K-D is not rinsed with fresh solvent when the ampule is removed. Quad and MDL studies have shown that this step is unnecessary.

Definitions
1. GPC – Gel Permeation Cleanup

2. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) – A sample of known composition analyzed with each batch of samples to demonstrate laboratory accuracy. The samples either naturally contain the analytes of interest or are clean samples fortified with known concentrations. Used to demonstrate laboratory accuracy. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

3. Matrix spike/matrix spike duplicate (MS/MSD) – A sample created by fortifying a second aliquot of a water or soil sample with some or all of the analytes of interest. The concentration added is known and compared to the amount recovered to determine percent recovery. Matrix spike recoveries provide information about the accuracy of the method in light of the matrix analyzed. A duplicate is a second aliquot of a sample that is treated identically to the original to determine precision of the test.

4. Surrogates – Organic compounds which are similar to the analytes of interest but are not naturally occurring in environmental samples. Surrogates are spiked into all standards and every

field and QC sample prior to extraction and analysis to provide information regarding the effects of the sample

Interferences

Method interferences may be caused by impurities in solvents, reagents, glassware, or other hardware used in sample processing. All glassware is rinsed with solvent before use and a method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.
Since the extracts are concentrated on a steam bath, caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or must be disposed of in the designated containers. These will then be transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the technicians training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control samples (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples are collected in wide-mouth glass jars with PTFE-lined lids and stored under refrigeration at 0º to 6º C, not frozen, prior to extraction. Samples must be extracted within 14 days of collection. Extracts are stored in the freezer at -10° to -15°C.

Apparatus and Equipment

1. MARS Xpress – CEM Corp. or equivalent

2. Kuderna-Danish (K-D) assembly with appropriate ampule for concentrating the solvent used during concentration

3. Steam bath, VWR/LLI Model #1127 or equivalent

4. N-Evap with nitrogen supply

5. Beakers – Stainless steel, assorted sizes

6. Pipettes – Class A, assorted sizes
7. Pipettes – Disposable

8. Balance – Capable of weighing to 0.01 g

9. Teflon®-wash bottles

10. Vials – Assorted sizes

11. Teflon®-boiling chips

12. Forceps

13. Scoop

14. TurboVap II concentration workstation w/appropriate concentration tubes – Zymark or equivalent

15. Funnels – stainless steel or Teflon®

16. Extraction vessels

17. Frits – Various

18. Sodium Sulfate Columns

19. glass vial inserts

Reagents and Standards

1. Hexane – Pesticide grade or equivalent. Fisher Optima grade or equivalent, stored in a FisherPak at room temperature for one year after receipt

2. Acetone – Pesticide grade or equivalent. Fisher Optima grade or equivalent, stored in a FisherPak at room temperature for one year after receipt

3. Methylene Chloride (CH_2Cl_2) – Pesticide grade or equivalent. Fisher Optima grade or equivalent, stored in a FisherPak at room temperature for one year after receipt

4. Sodium Sulfate (Na_2SO_4) – Reagent grade or equivalent. Bake at 400°C for a minimum of 4 hours in a shallow pan prior to use to remove organic contaminants. After baking, store in a glass jar at room temperature for up to 1 year.

5. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding Analysis #*0042, 1030, 6011, 7512, 10225, 10736, 10906, 12800, 13236*.

Preparation of Glassware

See ***T-OE-GEN-WI10864***

Calibration

Not applicable to this procedure

Procedure

1. Sample aliquot

a. If Sample Registration has pre-weighed the sample into a glass jar, add 5g of sodium sulfate, mix and proceed to step 2.

b. If the sample is not pre-weighed, weigh 30.0 - 30.5 g of sample into a labeled stainless steel beaker. (If matrix is tissue, weigh 15.0 - 15.5 g)

(1) Record the initial weight and any comments about the sample in the extraction log.

(2) Alternative sample weights may be used to meet certain reporting limits. However, if sample weight greater than 30.5 is used, the sample must be divided among multiple vessels and the extracts combined.

c. Process all tissues by Analysis #*2487* prior to extraction.

d. Add approximately 5 g of sodium sulfate to each sample and mix.

(1) If the sample has high water content or is a clay-like soil, add additional sodium sulfate until a free-flowing consistency is reached.

(2) Mix the sodium sulfate and sample until a free-flowing consistency is reached.

e. The Blank, LCS, and LCSD (if applicable) are prepared by filling a Teflon® extraction vessel with 35.0 - 35.5 g of sodium sulfate. (If sample matrix is tissue, use 15.0-15.5 g tilapia and add approximately 5 g sodium sulfate. Then mix.)

(1) For non-tissue samples: Record 30 g on the extraction log. (The sodium sulfate used for the QC samples is measured out as 35.0 grams to account for the 30 gram "sample" plus the 5 grams of sodium sulfate.)

2. Carefully place each sample into its clearly marked corresponding extraction vessel. A funnel is used to prevent spillage and loss of sample.

3. Use pipettes to add surrogate standards and spiking solutions.

a. Surrogates – 1.0 mL of SW846 surrogate is added to all samples (including QC).

b. Spiking solutions – 1.0 mL of PCB spiking solution is added to the LCS, LCSD if applicable, MS, and MSD.

c. This may change to accommodate specific client requirements.

d. See ***T-OE-GEN-WI10876*** for storage and handling of spikes.

4. Add 30 mL of 50% acetone in methylene chloride to each vessel.

5. Cap each vessel according to manufacturer's guidelines.

6. Invert each vessel to ensure mixing of sample and solvent.

7. Place the vessels into the carousel. When all samples are loaded, place the carousel into the microwave.

8. Run Program "LL1". See Table I for Instrument conditions. Verify that the run reached 100°C and document on the batchlog.

9. Uncap the cooled vessel. Pour the extract and sample into a column filled with approximately 10cm of sodium sulfate on top of a Kuderna-Danish (K-D) assembly containing a Teflon®-boiling chip. Rinse the vessel with approximately 10 mL of methylene chloride from a wash bottle and pour through salt column. Rinse the column with approximately 10 mL of methylene chloride.

10. Place a 3-ball Snyder column on the K-D set-up and concentrate over a steam bath which is at 85° to 99°C.

This steam bath temperature ensures concentration in a reasonable length of time.

If the sample requires GPC, skip step 11.

11. When the apparent volume is 3 to 5 mL, using a bottle-top dispenser, add approximately 50 mL of hexane directly to the KD through the Snyder column.

Do not allow the ampule to go dry since loss of analytes will result.

12. When the apparent volume again reaches 3 to 5 mL, remove from the steam bath and allow to cool for approximately 10 minutes until the K-D setup reaches ambient temperature. Remove the ampule and, using a wash bottle, adjust the final volume to exactly 10 mL with hexane in a calibrated ampule.

NOTE: If the sample requires GPC, N-Evap to approximately 1 mL, then adjust the final volume to exactly 10 mL with methylene chloride instead of hexane. N-Evap if necessary.

Mix thoroughly with a disposable pipette.

13. If the sample requires GPC, perform GPC cleanup following ***T-OE-PEST-WI11410*****.** When GPC cleanup is complete, concentrate the extract as described above including Step 11. However, in Step 12, adjust the final volume to exactly 5 mL with hexane instead of 10 mL.

14. Treat the 10 mL extract with sulfuric acid as described in ***T-OE-PEST-WI10281*****.**

15. Perform the copper and florisil cleanup on the extract as described in ***T-OE-PEST-WI10281*****.**

16. Bottle in three appropriately labeled crimp-top autosampler vials (one with an insert), and place the remaining extract in an appropriately labeled screw-cap vial.

17. All extracts are stored in the freezer.

Calculations
See analysis method.

Statistical Information/Method Performance
See analysis method.

Quality Assurance/Quality Control
A batch is defined as the samples to be extracted in any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

For each batch of samples extracted, a blank, LCS, MS, and MSD must be extracted. If insufficient volume of sample is available for MS/MSD, then an LCSD must be prepared instead.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Table I

Power:	1600W
Ramp Temperature:	100°C
Ramp Time:	30 minutes
Hold Time:	10 minutes
Cool Down Time:	20 minutes

T-OE-GEN-WI10864 Glassware Cleaning for Organic Extractions
T-OE-GEN-WI10876 Organic Extraction Standards Storage and Handling
T-OE-GEN-WI7154 Food and Tissue Preparation
T-OE-PEST-WI10281 Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-OE-PEST-WI11410 Pesticide Extract Cleanup Using GPC by Method 3640A
T-PEST-WI11972 Polychlorinated Biphenyls (PCBs) by Method 8082 in Solids and Wipes

End of document

Version history

Version	Approval	Revision information
7	03.DEC.2015	
8	17.APR.2018	

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E-06

Separatory Funnel Extraction by Method 3510C, 608 or 622 for Pesticides and PCBs in a Wastewater
(Rev. 19; 10/07/2016; T-OE-PEST-WI10920)

eurofins	Always check on-line for validity. **Separatory Funnel Extraction by Method 3510C, 608 or 622 for Pesticides and PCBs in a Wastewater**	Level: **Work Instruction**
Document number: **T-OE-PEST-WI10920**		
Old Reference: **1-P-QM-WI -9015079**		
Version: **19**		Organisation level: **5-Sub-BU**
Approved by: **EU5K** Effective Date **07-OCT-2016**	Document users: **5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Pest/PCB Waters, 6_EUUSLA_Pesticide Residue Analysis_All Management, 6_EUUSLA_Pesticide Residue Analysis_OC Pesticide C, 6_EUUSLA_Pesticide Residue Analysis_PCB Chemist**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID

Analysis DOD - 6654, 10241, 11112, 11113, 11114, 11116, 11117, 11118, 11119, 11120, 11121, 11123, 11126, 11960, 12026, 12822, 13086, 13093, 13183, 13187

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Revision Log

Revision: 19		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects current analysis numbers	Added Analysis #13634, 14134, 14166, 14169, 14184, 14186, 14188
Apparatus and Equipment	Reflects current practice	Added Automated Water Extraction Bench, Rapid-Vap Evaporator, and Rapid-Vap Evaporator Tube

Revision: 19		**Effective Date:** This version
Procedure 6.b. & 6.c.	Reflects current practice	Added surrogate and spike details for new analysis numbers
Procedure 11-19	Reflects current practice	Separate 1L extraction from 250 mL extraction and added new procedure for 250 mL extraction

Revision: 18		**Effective Date:** Apr 13, 2016
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1–P–QM-QMA-9017356	Removed revision logs up to the previous version
Apparatus and Equipment	Reflects current process	Added Turbo Vap tubes and Turbo Vap
Procedure 13	Reflects current process	Added 13.b for analyses that require Turbo Vap concentration
Procedure 18	Reflects current process	Added 18.b for analyses that require Turbo Vap concentration
Procedure 19	Reflects current process	Added 19.b, 19.c, and19.d to differentiate between which steps require Turbo Vap concentration and which steps require steam bath concentration.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3510C, Rev. 3, December 1996.
2. USEPA, 40 CFR Part 136, Appendix A, Method 608.
3. USEPA, 40 CFR Part 136, Appendix A, Method 622.
4. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0177, 0950, 0180, 1954	Pesticides in Water by Method 8081A using GC-ECD
Analysis #2257, 2253	Captan and Captafol by Method 8081A in Waters and Solids using GC-ECD
Analysis #5366, 10410, 10593, 12144, 13182, 13186	Organophosphorous Pesticides by Methods 8141A/8141B/622 in Aqueous Samples using GC-NPD
Analysis #6030, 10227	Polychlorinated Biphenyls (PCBs) by Method 608 or 8082 in Waters
Analysis #7572	Pesticides in Aqueous Samples by Method 608
Analysis #10589, 10647	Pesticides in Water by Method 8081B using GC-ECD
Analysis #10591, 13092	Analysis of Polychlorinated Biphenyls (PCBs) by 8082A in Aqueous Samples using GC-ECD

Analysis #12013, 12686	Low Level PCBs in Water by Method 8082/8082A using GC-ECD
1-P-QM-PRO-9015407	Pesticide Extract Cleanup Using Gel Permeation Chromatography
1-P-QM-PRO-9015475	Glassware Cleaning for Organic Extractions
1-P-QM-PRO-9015477	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
1-P-QM-PRO-9015490	Organic Extraction Standards Storage and Handling

Purpose
The purpose of this SOP is to provide clear instructions for performing the separatory funnel extraction procedure on samples that are to be analyzed for pesticides and PCBs.

Scope
This procedure is for the extraction of organochlorine and organophosphorous pesticides and PCBs from wastewaters.

Basic Principles
An aliquot of the sample is placed into a separatory funnel. The volume of sample extracted is adjusted (if appropriate) depending on the physical appearance of the sample and the volume sent for analysis. A surrogate standard is added to the sample to monitor recovery. The sample is then extracted with methylene chloride. The extract is dried, concentrated, and exchanged to hexane. Several cleanup procedures are available to eliminate matrix interferences before the sample is analyzed. They include sulfuric acid treatment, copper treatment, florisil, and Gel-Permeation Cleanup (GPC).

Reference Modifications
1. Surrogate and matrix spiking solutions are not added before the transfer to the extractor. For several reasons:

a. Samples must be poured from the amber bottles to determine the matrix and volume of sample to use for each extraction.

b. Many sample bottles have no headspace and there is no room to add surrogate to the sample in the bottle.

c. Due to the volume of samples extracted, a separate graduated cylinder for each sample is unrealistic.

d. To maintain consistency with all extractions, no samples are spiked in the bottle or graduated cylinders.

2. In Procedure Step 19 the joint of the KD is not rinsed with fresh solvent when the ampule is removed. Quad and MDL studies have shown that this step is unnecessary.

Interferences

Impurities in solvents, reagents, glassware, or other hardware used in sample processing interfere with the method. All glassware must be rinsed with solvent before use. A method blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Extracts are concentrated on a steam bath; caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are transferred to the lab wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the employees training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the extraction and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, or one blind sample.

Sample Collection, Preservation, and Handling

Samples are collected in amber glass bottles with PTFE-lined lids, preserved with sodium thiosulfate, and stored refrigerated at 0 - 6°C, not frozen. Sample extraction must be started within 7 days of collection. Extracts are stored frozen at ≤-10°C.

Apparatus and Equipment

1. Separatory funnel for extracting organic components from an aqueous matrix

2. Kuderna-Danish (K-D) assembly with appropriate ampule for extracting the solvent used during the extraction

3. Water bath – VWR/LLI Model #1127 or equivalent

4. Sodium sulfate column with extra course frit

5. Nitrogen evaporation (N-Evap) with nitrogen supply – Organomation Associates or equivalent

6. pH paper – Wide range

7. Automatic shaker – Glass Col or equivalent, capable of holding 2-L separatory funnels

8. Pipettes – Class A, assorted sizes

9. Graduated cylinders – Class A, assorted sizes

10. Pipettes – Disposable

11. Solvent dispenser – Brinkmann, adjustable or equivalent

12. Balance – Capable of weighing to 0.01 g

13. Centrifuge – Beckman GS-6 or equivalent

14. Micro-Snyder columns

15. Wash bottles – Teflon

16. Vials – Assorted sizes

17. Teflon boiling chips

18. Syringes - Assorted sizes

19. Micro-pipetter

20. Turbo Vap – Zymark Turbo Vap II concentration station or equivalent

21. Turbo Vap tubes- 250ml Zymark tubes or equivalent
22. Automated Water Extraction Bench – capable of holding separatory funnels for 250 mL extractions.

23. Rapid-Vap Evaporators – for concentration of 250 mL extractions

24. Rapid-Vap Evaporator Tubes – Class A

Reagents and Standards

1. Methylene chloride (CH_2Cl_2) – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

2. Acetone – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

3. Hexane – Pesticide grade or equivalent. Store at room temperature for up to 1 year.

4. 10N Sodium hydroxide (NaOH) – Lab Chem or equivalent. Store at room temperature. Follow manufacturer's expiration date.

5. Sulfuric acid (H_2SO_4) – ACS grade or equivalent. Store at room temperature. Follow manufacturer's expiration date.

6. Sodium Sulfate (Na_2SO_4) – Reagent grade or equivalent. Bake at approximately 400°C for a minimum of 4 hours in a shallow pan to remove organic contaminants. Store in a glass jar at room temperature for up to 1 year after baking.

7. Reagent water – water in which an interferent is not observed at or above the reporting limit for parameters of interest. In general, the reagent water supplied at the taps in the laboratory meets this criterion. If the reagent water does not meet the requirements, see your supervisor for further instructions.

8. Extraction fluid – Prepared and delivered by the leachate department. Store refrigerated at 0-6°C, not frozen, in a glass container with a PTFE-lined lid.

9. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding analytical Analysis #0177, 0950, 0180, 1954,14134; Analysis #2257, 2253;
Analysis #5366 10410, 10593, 12144, 13182, 13186; Analysis #6030, 10227, 14169, 14188; Analysis #7572, 13634; Analysis #10589, 10647, 14166; analysis 10591, 13092, 14184, 14186 ; and Analysis #12013, 12686.

Calibration

Not applicable to this procedure.

Preparation of Glassware

See SOP 1-P-QM-PRO-9015475.

Procedure

1. Determine the volume of sample to be used for each extraction. Typically, this is 1L for routine extractions or 250mL for 250mL extractions.

1 Liter Analyses: 0177, 0180, 1954, 2257, 2253, 5366, 10410, 10593, 12144, 13182, 13186, 6030, 10227, 7572, 10589, 10647, 10591, 13092, 12013, 12686

250 mL Analyses: 13634, 14134, 14166, 14169, 14184, 14186, 14188

a. If uncertain of the volume to extract for any sample, ask your supervisor.

b. Use one full bottle for all analysis scans (with the exception of scan #0950) unless the matrix is poor (thick, lots of sediment, extremely foul odor).

(1) If using reduced volume due to matrix, take as much as possible while trying to minimize matrix problems, document why the reduced volume was used.

(2) Reduced volume aliquots due to matrix are typically are 500, 200, or 100 mL.

c. For analysis 0950:

(1) If the sample bottle contains at least 500 mL of sample, measure 200 mL.

(2) If the sample bottle contains <500 mL of sample, use 1/2 of the available volume or 10 mL, whichever is greater.

(3) If a matrix spike (MS) or MS/matrix spike duplicate (MSD) is required for the sample, use 200 mL each.

d. The background, MS, and MSD are performed on three separate aliquots of a field sample.

2. Prepare the blank, laboratory control sample (LCS), laboratory control sample duplicate (LCSD) (if applicable) with 1 L of reagent water (or 250mL for 250 mL extractions) measured into the separatory funnel.

Exception: For Analysis #0950 - The blank, LCS, and LCSD (if applicable) are prepared using 200 mL of extraction fluid measured into the separatory funnel.

3. For samples using 1 entire bottle:

a. Etch the outside of the bottle with a scriber at the meniscus.

b. Shake the bottle vigorously and then pour the contents into a separatory funnel.

4. For all samples requiring a specified volume:

a. Shake each bottle vigorously.

b. Use a clean graduated cylinder to measure the desired volume.

c. Use a wash bottle to rinse the graduated cylinder with methylene chloride and add the rinsate to the separatory funnel.

d. If <1000 mL (or <250mL for 250mL extractions) of sample is used, use a graduated cylinder to add enough reagent water to bring the volume in the extractor to 1−L (or 250mL for 250mL extractions).

5. Record any comments about the sample and the initial volume, if known at this time, on the extraction sheet.

6. Use pipettes to add surrogate standards and spiking solutions to the aqueous sample in the separatory funnel.

a. Be certain the standard drips directly into the aqueous sample without touching the glass side of the separatory funnel to avoid poor recoveries.

b. Surrogates – Surrogates are added to all samples, blanks, and spikes.

The type of surrogate is determined by the analysis scan number. Typically they are as follows:

(1) For analyses 0177, 0180, 0950, 1954, 2257, 6030, 7572, 10589, 10591, 13092, 10647, 13092 – 1.0 mL SW−846 Surrogate.

(2) For analysis 5366, 10593, 10410, 12144, 13182, 13186 – 1.0 mL NP Surrogate

(3) For Analysis #12013, 12686 – 0.1 mL of SW-846 Surrogate

(4) For Analysis #10227 – If entered for prep 11117 use 1.0 mL of SW846 Surrogate. If entered for prep 13086 use 1.0 mL of 2mL SW846 Surrogate.

(5) For Analyses #13634, 14134, 14166, 14169, 14184, 14186, 14188 – Use 1.0 mL of Mini SW846 Surrogate

c. Spiking Solutions – Spiking solutions are added to the LCS, LCSD (if applicable), MS, and MSD.

(1) The type of spike is determined by an analysis number. Typically they are as follows:

(a) For analysis 6030, 10591, 13092 – 1.0 mL PCB Spike

(b) For analysis 10227 – If entered with prep 11117 use 1.0 mL of PCB spike. If entered with prep 13086 use 1.0 mL of 2mL PCB spike.

(c) For analysis 7572, 10589, 0177 – 1.0 mL SW-846 Spike

(d) For analysis 0950 & 10647 – 1.0 mL TCLP Pesticides Spike regardless of initial volume

(e) For analysis 5366 – 1.0 mL Triazine Herb Spike

(f) For analyses 10593 and 10410 – 1.0 mL OPPA Spike

(g) For analysis 2257 – 1.0 mL Captan/Captafol Spike

(h) For analysis 0180 – 1.0 mL of Chlordane spike

(i) For analysis 1954 – 1.0 mL of SW846 spike and an additional LCS/LCSD with 1.0 mL of Alaclor

(j) For analysis 12013 – 0.1 mL of PCB Spike

(k) For Analysis 13093 – 1.0 mL of PCB Spike and prepare a separate LCS/LCSD using 1.0 mL of 5442 Arochlor Spike

(l) For Analysis 13182 and 13186 – 1.0mL of Appendix IX Water Spike (or 0.25 mL of Appendix IX water Spike for 250mL extractions).

(m) For analysis 13634, 14134, 14166 – 0.25 mL of SW846 Spike

(n) For analysis 14169, 14184, 14188 – 1.0 mL of Mini PCB Spike

(o) For analysis 14186 – 1.0 mL of Mini PCB Spike and prepare a separate LCS/LCSD using 1.0 mL of Mini 5442 Arochlor Spike

(2) Spike details can be found in the corresponding analytical SOP(s)

(3) This is changed to accommodate specific-client requirements, if appropriate.

(4) If a sample requires any special compounds in addition to the standard list, an appropriate spike containing those compounds is added at this time.

(5) See SOP 1-P-QM-PRO-9015490 for storage and handling of spikes.

7. Measure and record the pH of the sample using wide-range pH paper.

a. If necessary, adjust the pH to between 5 and 9 using 10N NaOH (to bring up the pH) or concentrated H_2SO_4 (to lower the pH).

b. To adjust the pH, add a few drops of the appropriate solution with a disposable pipette, shake the separatory funnel and re-check the pH.

c. If the pH now falls between 5 and 9, record the adjusted pH on the extraction sheet.

d. If the pH is still out of range, continue adding small aliquots of base or acid, shaking and checking the pH until it is within the specified range.

e. Record the adjusted pH.

NOTE: Any samples from North Carolina require that the volume of acid or base added to the sample to adjust the pH to be recorded on the extraction sheet.

8. If the original sample bottle is empty:

a. Use a solvent pump to measure 60 mL (15 mL for 250 mL extractions) of methylene chloride. Add the methylene chloride to the sample bottle. Then cap the bottle and invert several times. Add the solvent to the separatory funnel.

b. Fill the bottle to the marked level with water and transfer the water to a graduated cylinder to determine the initial volume.

c. Alternatively, for all analyses **except Analysis #7572, and analysis 6030 (Method 608)** weigh the empty bottle and tare the balance.

(1) Fill the bottle to the marked level with water and place the bottle onto the tared balance.

(2) This weight rounded to a whole number is the initial sample volume.

d. Record the initial volume on the extraction sheet.

9. If the sample container is not empty, measure 60 mL (15 mL for 250mL extractions) of methylene chloride and add the solvent directly to the separatory funnel.

10. Cap the funnel, invert it, and vent immediately.

11. For 1 Liter Analyses: Place the sample on the automatic shaker and shake at the designated speed for 2 minutes with the stopcocks closed.

For 250 mL Analyses: Separatory funnels remain on the Automated Water Extraction Bench. Press the green “start” button to lower the hood and activate the Automated Water Extraction Bench. The bench will tumble for 2 minutes. The sash will rise after the samples have tumbled.

NOTE: Shaker speeds vary greatly between instruments so the proper setting is marked on each.

12. For 1 Liter Analyses: Place the separatory funnel on the rack and allow it to sit undisturbed for approximately 10 minutes. For 250 mL Analyses: the separatory funnel will sit undisturbed on the Automated Water Extraction Bench.

a. The time required for extracts to set undisturbed is based upon visual confirmation that the layers are adequately separated. Additional time may be necessary for samples with unusually high density (i.e. high salt content).

b. If an emulsion forms and is greater than 1/3 of the volume of the solvent layer, mechanical techniques such as stirring and centrifugation must be employed to complete the separation.

13 Removing the Solvent Layer.

a. For 1 Liter analyses (except 5366, 10593, 10410, 12144, 13182, and 13186) : Remove the solvent layer by opening the stopcock and collect the solvent in a metal beaker then pour through approximately 10cm of sodium sulfate into a K-D apparatus containing a Teflon boiling chip.

b. For analyses 5366, 10593, 10410, 12144, 13182, and 13186, remove the solvent layer by opening the stopcock and collecting the solvent in a metal beaker. The solvent layer is then poured into a sodium sulfate column and collected in a Turbo Vap tube.

c. For 250 mL analyses: drain the solvent layer directly into the sodium sulfate column containing approximately 5cm of sodium sulfate. This drains into a Rapid-Vap Evaporator Tube.

NOTE: Do not use more than the recommended quantity of sodium sulfate and take care not to transfer water into the column. Excessive sodium sulfate and/or water in the column results in low aldrin recovery.

14. Use a solvent pump to add 60 mL (15mL for 250mL extractions) of methylene chloride to the separatory funnel and repeat Procedure Steps 10 through 13, venting only as necessary.

15. Again, use a solvent pump to add 60 mL (15 mL for 250mL extractions) of methylene chloride to the separatory funnel and repeat Procedure Steps 10 through 13, venting only as necessary.

16. For 1L extractions only, rinse the metal beaker that was used for solvent collection with approximately 20 mL of methylene chloride. Pour into salt column.

17. Rinse salt column with approximately 20 mL (5mL for 250mL extractions) of methylene chloride. Use a hand held bulb to squeeze through any excess methylene chloride.

18. Concentrating the Extract.

a. For 1 Liter Analyses: Attach a 3−ball Snyder column to the K−D, wet with solvent, and concentrate the extract to approximately 3 to 5 mL on a steam bath at 85° to 99°C. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. This steam bath temperature ensures concentration in a reasonable length of time.

b. For 250 mL Analyses: Place the Evaporator Tube containing the solvent extract into the pre-heated (approximately 80°C) Rapid-Vap Evaporator. Set the timer for 15 minutes and speed at 75% (the speed is the rotation within the unit while the sample is evaporating). Confirm that the nitrogen is set at 15 psi (this is adjusted on the regulator attached to the nitrogen line). Allow samples to concentrate to between 1-2mL.

Note: The temperature, timer, and speed settings can be adjusted as necessary to accommodate difficult sample matrices, etc.

c. For analyses 5366, 10593, 10410, 12144, 13182, and 13186, place the Turbo Vap tubes in a Turbo Vap with the temperature set between 45°and 50°C.

To avoid loss of analytes do not allow the ampule or Turbo Vap tube to go dry

19. Allow the sample to cool for 10 minutes.

a. If the sample does not require GPC and a steam bath concentration or Rapid – Vap evaporation is required:

(1) Use a graduated cylinder to add approximately 50 mL of hexane directly to the K−D through the Snyder column for 1 Liter analyses or directly to the Rapid-Vap Evaporator Tube for 250 mL analyses.

(2) For 1 Liter Analyses: Concentrate until a volume of 3 to 5 mL is achieved. For 250 mL Analyses: Concentrate to around 1 mL.

(3) Remove the sample from the bath (or Rapid-Vap Evaporator) and allow the sample to cool for 10 minutes.

(4) For 1 Liter Analyses: Remove the ampule and use a wash bottle to adjust the final volume to exactly 10.0 mL with hexane in a calibrated ampule. For 250 mL Analyses: Bring to final volume 2mL in the Class A Evaporator Tube by rinsing with hexane.

Exception:

(a) If samples are scheduled for Prep 12026, remove the ampule and place on an N -evap until the volume is below 5 mL. Adjust the final volume to 5 mL with hexane in a calibrated ampule.

(b) If samples are scheduled for Prep 13093,, remove the ampule and place on an N-evap until the volume is below 2mL. Adjust the final volume to 2mL with Hexane in a calibrated ampule.

(5) Mix extract thoroughly with a disposable pipette.

b. if the sample does not require GPC and a Turbo Vap concentration is required.

(1) Use a graduated cylinder to add approximately 50ml of hexane directly to the Turbo Vap tube.

(2) Concentrate until a volume of <2 ml is achieved.

(3) Remove the sample from the Turbo Vap. Quantitatively transfer the extract with hexane into a calibrated ampule and bring to a final of 10ml for 1liter preps and to 2ml for 250ml preps.

(4) Mix thoroughly with a disposable pipette.

c. If the sample requires GPC and a steam bath concentration or Rapid-Vap Evaporation is required.

(1) For 1 Liter Analyses: Remove the ampule and adjust the final volume to exactly 10.0 mL with methylene chloride. For 250 mL Analyses: Quantitatively transfer the sample from the Evaporator Tube and bring to FV 10.0 mL with methylene chloride using a Class A volumetric.

(2) Mix thoroughly with a disposable pipette.

(3) Perform GPC cleanup following SOP 1-P-QM-PRO-9015407.

(4) Once the GPC cleanup is completed, place the extract in a K-D containing a Teflon boiling chip.

(5) Attach a 3-ball Snyder column to the K-D, wet with solvent, and concentrate the extract to approximately 3 to 5 mL on a steam bath at 85° to 99°C. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood.

(6) Allow the sample to cool for 10 minutes.

(7) Use a graduated cylinder to add approximately 50 mL of hexane directly to the K-D through the Snyder column.

(8) Concentrate until a volume of 3 to 5 mL is achieved.

(9) Remove the sample from the bath and allow the sample to cool for 10 minutes.

(10) Remove the ampule and use a wash bottle to adjust the final volume to exactly 5.0 mL with hexane.

(11) Mix thoroughly with a disposable pipette.

d. If the sample requires GPC and Turbo Vap concentration is required.

(1) Remove the Turbo Vap tube from the Turbo Vap. Quantitatively transfer the extract into a calibrated ampule and bring to a final volume of 10ml with methylene chloride.

(2) Mix thoroughly with a disposable pipette.

(3) Perform GPC cleanup following SOP 1-P-QM-PRO-9015407.

(4) Once the GPC cleanup is completed, place the extract in a Turbo Vap tube.

(5) Place the Turbo Vap tube in a Turbo Vap with the temperature set between 45° to 50°C. Concentrate the extract to approximately 3 to 5ml.

(6) Use a graduated cylinder to add approximately 50ml of hexane directly into the Turbo Vap tube and concentrate to approximately 2ml.

(7) Remove the sample from the Turbo Vap. Quantitatively transfer the extract into a calibrated ampule using hexane to bring to a final volume of 5ml.

20. Complete Procedure Steps 21 through 24 as needed.

21. If the extract is scheduled for analysis 6030, 10227, 12013, 14169, 14184, 14186, 14188 treat the extract with sulfuric acid as described in SOP 1-P-QM-PRO-9015477.

22. If the extract is scheduled for analysis 0177, 7572, 10589, 13634, 14134, 14166 florisil the sample 2 mL to 2 mL as described in the Pesticide Florisil section of SOP 1-P-QM-PRO-9015477.

Pesticide florisil is also being performed for analysis 0950 and 2257 if required due to matrix or client request.

23. If the extract is scheduled for analysis 6030, 10227, 12013, 14169, 14184, 14186, 14188 florisil the extract following the PCB Florisil section of SOP 1-P-QM-PRO-9015477.

24. If the extract has a sulfur odor, perform copper cleanup of the extract as described in SOP 1-P-QM-PRO-9015477 for analysis 6030 only. Other samples are cleaned up with GPC.

Calculations

See analysis method as listed in the Cross Reference section.

Statistical Information/Method Performance

See analysis method as listed in the Cross Reference section.

Quality Assurance/Quality Control

A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. Exception: For analyses referencing Methods 608 or 622 (i.e. 7572, 6030, 11119, 11123,13634, 14188) batches cannot exceed 10 field samples.

For each batch of samples extracted a blank, an LCS, an MS and an MSD must be extracted. For method 608, the laboratory must, on an ongoing basis, spike at least 10% of the samples, if enough sample volume is received, to assess accuracy. If there is limited sample preventing

the preparation of the MS/MSD, an LCSD must be prepared instead. If the batch contains only field or equipment blank samples, the LCS/LCSD QC pairing must be used.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed instead.

End of document

Version history

Version	Approval	Revision information
19	07.OCT.2016	

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E-07

Polychlorinated Biphenyls (PCBs) in Solid Samples by 8082A Using GC-ECD (Rev. 9; 03/29/2018; T-PEST-WI10004)

LIMS ID

Analysis - 10592, 10885, 12718, 13099, 13219, 13713

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Revision Log
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Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
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Reagents and Standards
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Calibration
Procedure
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Appendix I

Revision Log

Revision 9		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version

Revision 9	**Effective Date:**	This version
Reference	Reflects current references	Added 8000D
Cross Reference	Updated references to D4	Changed from ETQ reference to D4 throughout document
Interferences	Enhancement	Added information on DDD,DDT, and DDE interference
Sample Collection, Preservation, and Handling	Reflects method hold time	Changed hold time to none recommended
Reagents and Standards 5.	Clarification	Removed sentence on 2 different sources for ICAL and matrix spike
Reagents and standards	Correction	Updated surrogate stock used for IBLK.
Calibration	Reflects current sequence	Added AR166, AR486, and AR546 Changed CCVs from level 3 to level 4.
Calibration 2.a.(4)	Clarification	Added 1248
Calibration 5.	Clarification	Added wording on using each individual peak choosen in the pattern for calculating ave CF.
Statistical Information/Method Performance	Reflects current process	Changed wording to similar verbiage as other department SOPs. added LLOQ requirement
Appendix I	Reflects current sequence	Added AR166, R486 and AR546

Revision: 8	**Effective Date:**	Aug 26, 2016
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Cross Reference	References not needed	Removed 1-P-QM-QMA-9015390 and 1-P-QM-QMA-9017309
Scope	Reflects current Aroclors	Updated table to include Aroclor 1262 and 1268 Added 1262 and 1268 to analysis scan 13099 in the table
Basic Principles	Old reference not needed	Removed old reference number SOP-OE-004
Reference Modification	Reflects the current reference method	Updated reference to 8082A
Definition	Common laboratory terms defined in higher level documents	Removed definitions
Interferences	Old reference not needed.	Removed old reference number SOP-OE-004
Personnel Training and Qualifications	Reference not needed	Removed reference 1-P-QM-QMA-9015390.

Revision: 8	**Effective Date:** Aug 26, 2016	
Sample Collection, Preservation, and Handling	Reflects current storage condition for extracts	Added temperature storage condition for extracts.
Apparatus and Equipment	Reflects current instrumentation in use	Updated to HP7890
Reagents and Standards	Reflects current standard solution storage condition	Updated standard solution temperature storage condition from -10 to -15°C to ≤-10°C
Reagents and Standards 18.d	Reflects current MS and SS spike solutions	Updated to the current spike solution names.
Extraction	Reflects current scans	Added Analysis #13100
Calibration	Reflects current process Reflects current analysis sequence Reflects current naming convention	Removed changing the septa prior to calibrating. Updated sequence to include running the EVAL. Renumbered the sequence. Updated name from AR32X to AR323
Calibration 2.a	Reflects current standard code	Updated name from AR32X to AR323 and renumbered section.
Calibration 5.e.	Reflects current process Old references not needed	Added North Carolina criteria for linear fit. Removed old reference numbers SOP-PP-031, SOP−PP-013, and SOP-PP-032
Procedure 2.a	Old reference not needed	Removed old reference number SOP-PP-011
Procedure 4	Reflects current practice	Added hexane as the solvent used for dilutions
Calculation	Old reference not needed	Removed old reference number SOP-PP-040
Statistical Information/Method Performance	Reference not needed	Removed reference 1−P−QM−QMA−9017309
Quality Assurance/Quality Control	Clarification Old reference not needed	Moved QC acceptance limits statement from Statistical Information/Method Performance section to this section. Removed old reference number SOP-PP-002
Appendix I C.	Reflects current sequence Reflects current MS and SS spikes	Added EVAL and renumbered sequence Updated names of parent solution and solvent used for each spike preparation

Reference

1. *Test Methods for Evaluating Solid Waste*, SW-846, Method 8082A, February 2007.

2. Determinative Chromotographic Separations, SW-846, Method 8000D, July 2014

3 *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0819, 11128, 11132, 11135	Ultrasonic Extraction for PCBs in a Solid Matrix by Method 3550C
Analysis #10497, 11140, 13100	Microwave Extraction Method 3546 for PCBs in a Solid Matrix
T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-PEST-WI10011	QC Data Acceptability and Corrective Action
T-PEST-WI9954	Interpretation of Chromatographic Data
T-PEST-WI10007	Preventative and Corrective GC Maintenance
T-PEST-WI9980	Monitoring QC Data Acceptance Limits
T-PEST-WI10016	Setting Up Single Component Initial Calibrations
T-PEST-WI10022	Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses

Scope

This method is used for identifying and quantitating the following PCBs in solid samples and wipes using SW846 8082A:

Compound	Soil LOQ (µg/kg)	Wipes LOQ (µg)	Tissue LOQ (µg/kg)
Aroclor 1016	17	0.5	34
Aroclor 1221	17	0.5	34
Aroclor 1232	17	0.5	34
Aroclor 1242	17	0.5	34
Aroclor 1248	17	0.5	34
Aroclor 1254	17	0.5	34
Aroclor 1260	17	0.5	34
Aroclor 1262	17	0.5	34
Aroclor 1268	17	0.5	34

Aroclors 5422, 5432, and 5460 can be analyzed upon request of the client. See Appendix I for GC operating conditions and calibration information.

Compound	Soil LOQ (µg/kg)
Aroclor 5422	33
Aroclor 5432	33
Aroclor 5460	33

Limits of Quantitiation (LOQs) are based on statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

Analysis LIMS scan	Extraction LIMS scan	Description (targets)
10592	11132 sonication	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262, 1268
10885	10497 microwave	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262, 1268
13099	13100 microwave	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, 1268 Aroclor 5432, 5442, 5460
12718 wipes	10497 microwave	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262,1268
13219 tissue	11128 sonication	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262,1268
13713 tissue	10497 microwave	Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260 Aroclor 1262,1268

Basic Principles

A solid sample or entire wipe is extracted using sonic probe with 1:1 methylene chloride:acetone, or microwave extraction with hexane. The extract is dried, concentrated, and exchanged to hexane. A florisil and sulfuric acid cleanup is utilized to reduce any matrix interferences. The PCBs are then identified and quantitated using gas chromatography (GC) with electron capture detector (ECD). Copper cleanup may also be employed to reduce elemental sulfur or other matrix interferences which introduce large, unresolvable peaks into the chromatogram. Refer to *T-OE-PEST-WI10281* for details on each cleanup procedure.

Solid matrices other than soil can also be analyzed as long as the sample can be handled through the extraction technique. Typically solids are reduced to small pieces for extracting (concrete,

wood, other plant material etc). Tissue samples are ground and homogenized prior to extracting. Tissue samples may be whole fish, filets, or other miscellaneous species (usually aquatic but not necessarily). Wipe samples are analyzed by taking the entire wipe sample and any solvent in the sample container.

Reference Modifications

Gas Chromatography conditions are different than those listed in Method 8082A however, all QC criteria are met.

Interferences

Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.

Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.

An electron capture detector (ECD) is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur.

Extracts may require further cleanup by sulfuric acid, florisil, or copper if interferents such as oxygenated organics, unsaturated organics, and elemental sulfur are present. Refer to *T-OE-PEST -WI10281*
for details on each cleanup procedure.

Common single component pesicides such as DDT, DDD, and DDE may cause interference with the aroclor pattern. To ensure that the analyst is aware of this, a standard containing DDE/DDD/DDT will be run with each initial calibration.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Safety glasses must be worn around the GC where solvents and sample extracts are handled.

GC vials are disposed of in the designated lab container and subsequently lab packed for final disposal. All solvent waste is placed in designated containers in the laboratory then taken to the lab−wide facility by personnel trained in hazardous waste disposal.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs.

Sample Collection, Preservation, and Handling

Samples are collected in wide−mouth glass containers with Teflon™−lined caps and kept cool at 0° to 6°C, not frozen.

PCBs have no maximum recommended holding time for the extraction. Some agencies or projects may require a specific hold time for the extraction. If the hold time is specified in the project, this hold time must be followed. Sample analysis must be performed with 40 days of extraction. Extracts are stored in the freezer at ≤-10°C

Apparatus and Equipment

1. HP 7890 gas chromatograph equipped with an electron capture detector or equivalent

2. Columns:

   a. Phenomonex MultiRes I – 30 m × 0.32 mm × 0.5 µm or equivalent

   b. Phenomonex MultiRes II – 30 m × 0.32 mm × .25 µm or equivalent

   c. Alternatively, Restek columns can be used.

3. Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent. Chrom Perfect® is a data system capable of storing and reintegrating chromatographic data and determining peak areas using a forced baseline, area summation, baseline projection, and performing baseline compensation as required.

4. Various sizes of Class A volumetric flasks, pipettes, and syringes

Reagents and Standards

A. Reagents

1. Hexane for autosampler rinse vials. Stored at room temperature.

2. UPC (Ultra pure carrier) helium for carrier gas

3. UPC nitrogen for detector make-up gas

4. UPC hydrogen for carrier, either bottled or from a generator

B. Standards

1. All standards are prepared using Class A volumetric pipettes, syringes, and flasks.

2. An analytical balance is used to measure all weights.

3. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.

4. All prepared standard solutions are stored at ≤-10°C in labeled containers or vessels.

5. Aroclor 1016 & 1260 stock – Restek #32039 at 1,000,000 ppb in isooctane or equivalent.

   Prepare a ten−fold dilution for an intermediate.

6. Aroclor 1016 ICV Stock – Accustandard Cat# C-2165-H-10X at 1,000,000 ppb in Hexane.

7. Aroclor 1260 ICV Stock - Accustandard Cat# C-2605-H-10X at 1,000,000 ppb in Hexane.

8. Aroclor 1221 stock – Restek #32007 at 100,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.

9. Aroclor 1232 stock – Restek #32008 at 100,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.

10. Aroclor 1242 stock – Restek #32009 at 100,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.

11. Aroclor 1248 stock – Restek #362010 at 1,000,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source

    Prepare an intermediate by diluting 1 mL to 10 mL of hexane. .

12. Aroclor 1254 stock – Restek #32011 at 1,000,000 ppb in isooctane or equivalent. Two lots are kept on hand to provide a second source.

    Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

13. Surrogate stock (SS) – Supelco #861284 containing DCB/TCX at 200,000 ppb in acetone or equivalent.

    Prepare an intermediate by diluting 1.0 mL to 25 mL of hexane for AR1016/1260, AR1254, AR1248.

14. 1016/1260 Matrix spike (MS) stock – Restek #32039 at 1,000,000 ppb in hexane or equivalent.

15. Aroclor 1262 stock – Restek #32409 at 1,000,000 ppb in hexane or equivalent.

Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

16. Aroclor 1268 stock – Restek #32410 at 1,000,000 ppb in hexane or equivalent.

Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

17. Instrument Blank (IBLK) – Surrogate Stock (SS) – Restek #32000 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 2000,000 ppb in acetone or equivalent.

18. Prepare working standards using the electronic standard database as a guide.

a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. AR161), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

d. The scheme for preparing the matrix and surrogate spiking solutions used in the extraction process are listed below.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
PCB Spike	Aroclor 1016/1260 MS Stock	1.25	250	Acetone	PCB Spike
SS	Pest Surr Stock	1.5	1000	methanol	SW-846 Water surrogate – identical to

					that prepared for Pest/PCB analyses

19. An initial calibration verification standard must also be prepared at a concentration at the mid-point of the calibration for 1016, 1260,1248 and1254 using a stock solution purchased from a different source other than that used for the calibration standards.

20. Additional standards and preparations are listed in Appendix I for Aroclor 5442, 5432, and 5460 (PCT analysis).

Extraction

See Organic Extraction Analysis #0819, 11128, 11132, 11135 or Analysis #10497, 11140, 13100

Gas Chromatographic Conditions

The conditions listed are usually the optimum operating conditions but can vary to improve the sensitivity, linearity, and overall chromatography or shorten run times on each GC system.

Detector	ECD
Detector Temperature	330°C
Oven Temperature	110°C 40°C/min to 250°C 20°C/min to 280°C 30°C/min. to 330°C Hold until DCB elutes ~ 2 min.
Carrier	Hydrogen at 12 psi, 5ml/min constant flow (Can be substituted with Helium)
Makeup gas	N2 at 30 mL/min. for Varian GCs N2 at 55 mL/min. for HP GCs
Injection size	1 µL, direct injection
Injection Temperature	225°C

A Merlin microseal may be used in place of a traditional septum.

Calibration

1. Fill the autosampler rinse vials with clean solvent or replace vials which appear dirty.

2. Prepare a sequence using the following suggested order of injections:
    1. Conditioner
    2. IBLK
    3. EVAL
    4. AR161
    5. AR162
    6. AR163
    7. AR164
    8. AR165
    9. AR166
    10. AR481
    11. AR482
    12. AR483
    13 AR484
    14. AR485
    15. AR486
    16. AR541
    17. AR542
    18. AR543
    19. AR544
    20. AR545
    21. AR546
    22. AR214
    23. AR324
    24. AR424
    25. AR624
    26. AR684
    27 AR16xx
    28. MD16 (MDL)
    29. IC16x (ICV)
    30. IC48x (ICV)
    31. IC54x (ICV)
    32. Blank
    33. LCS
    34. 1234567
    35. 1234567ms
    36. 1234567msd
    37 – 47. Continue running samples
    48. AR164 (CCV)
    49. IBLK

a. For routine PCBs:

(1) The AR21, AR32, and AR42 are level 4's in the cal and are entered at AR214, AR324, and AR424 in the cal.

(2) AR62 and AR68 are both level 4s and are listed as AR624 and AR684 in the sequences.

(3) A single point of Aroclor 1016 is analyzed with the calibration to aid in better pattern recognition in the samples. Aroclor 1016 and 1242 have similar patterns with the exception of the smaller trailing peaks at the end of the Aroclor 1242 pattern. This is identified in the sequence as AR16xx.

(4) A DDT/Endrin breakdown standard is run with each ICAL in order to provide the retention times for p,p-DDE, p,p-DDD, and p,p-DDT which may interfere with the pattern for aroclor 1248, 1254 and 1260.

b. See Appendix I for a sequence example when the analysis of PCTs (Aroclors 5442, 5432, and 5460) is requested.

3. Inject conditioner to prime the system.

a. The conditioner injection is usually a standard or sample which has already been injected.

b. It is used to prime the system and is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

c. Hexane blanks may also be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system.

4. An instrument blank (IBLK) is always run prior to a new calibration to confirm that the instrument is free of background noise or contamination.

IBLK may also be run with the continuing check standards - this is optional but frequently requested for projects.

5. Initial Calibration (ICAL)

a. Calibrate for the aroclors using the six levels of 1016, 1260, 1248, and 1254 (calibration range of 50 µg/L through 1000 µg/L) and using the single point for 1221, 1232, 1242, and 1262 and 1268 when needed (200 µg/L standard used).

b. As an option, 1248 and 1254 may use a single point calibration.

c. An external standard calibration based on the average calibration factor (AVG CF)for each individual peak chosen in the pattern is used for quantitation where the %RSD is ≤20%.

d. The surrogate standards are calibrated using the AR16 levels and the calibration is also performed using AVG CF unless the %RSD is >20%.

e. When the % RSD criteria is not met (i.e.> 20%), a linear calibration curve is used.

(1) The curve must meet a correlation coefficient of 0.99 to be a valid fit.

(2) Extrapolate or force to zero is not allowed. Set the zero to ignore.

(3) See *T-PEST-WI10016* for more details.

(4) North Carolina and 8000D requires calculation of % error for any linear fits. The criteria for % error of standard levels at or below the LOQ is +/- 50% . The criteria for % error of all other standard levels is +/-30%.

%error = ((true – calc)/true) x 100

f. If the %RSD criteria or 0.99 curve coefficient cannot be met:

(1) Inspect the data points to see if one or more calibration levels may have concentrated due to solvent evaporation or degraded over time.

(2) Reinject or remake the standard if this is the cause.

(3) Perform instrument maintenance as needed.

(4) See *T-PEST-WI10007* for troubleshooting linearity problems.

A quadratic fit may not be used for South Carolina samples.

g. Set up the aroclor calibration data in a custom program under Datalog.

(1) The retention times of the peaks to use for identifying and quantifying the aroclors are entered into the calibration file along with the corresponding peak heights and concentrations (in µg/L).

(2) See *T-PEST-WI10022* for details.

h. Ensure the surrogate peaks in the standards are labeled properly.

i. Set the scaling of chromatograms so that the size of the peaks of interest for each aroclor are approximately 2 to 3 mm in height at the concentration of the method detection limit (MDL).

(1) Ensure all peaks in the MDL standard are integrated.

(2) By running the1016/1260 MDL standard, the majority of peaks for all aroclors should be represented.

6. Initial Calibration Verification (ICV)

a. Verify the calibration curves using the ICV mixtures injected directly after the full ICAL.

b. The % difference of the concentrations of each individual peak used to calculate the Aroclor must be within 20% difference of the nominal concentration.

7. Continuing Calibration Verification (CCV)

a. Calibration verification is performed after each set of twenty injections (samples, QC, blanks, etc.) or 12 hours, whichever comes first.

b. Use AR16 to evaluate the calibration of the aroclors (or other aroclors as requested for particular clients or projects).

c. The concentration quantitated for each individual peak used to calculate the Aroclor in the continuing calibration verification standards must be within 20% difference (%D) of the nominal concentration for each peak/surrogate.

d. Samples must be bracketed by compliant standards.

(1) Exception: If standard following a sample is outside the ±20% but exhibits increasing response, the samples before it do not have to be reinjected if the target analytes are not detected. A comment must be added to the analytical report.

(2) If two consecutive/sequential CCVs fail, corrective action must be taken, which can include performing injection port maintenance, baking the instrument or other types of maintenance. Two consecutive/sequential CCVs must meet the criteria or an initial calibration must be performed before sample analysis can continue.

e. If confirmation of target analytes is needed, then the second column should meet the 20% continuing calibration criteria, as well as all initial calibration criteria. if either of these are not met, a comment must be added to the analytical report.

8. The instrument blank (IBLK) is injected after each set of continuing calibration verification when requested.

a. It must be evaluated as a water matrix against the water MDL/LOQs.

b. The IBLK must not have any target compounds above the reporting limits.

(1) If a target analyte is detected in the IBLK, any associated samples with a detection for that same target must be evaluated.

(2) Unless the concentration in the sample is more than 10x the IBLK value, the sample must be injected after another compliant IBLK.

(3) Instrument maintenance, i.e., baking the system or injection port maintenance is usually necessary to clean up the instrument.

9. Retention time (RT) windows

a. Established as 3× the standard deviation determined over 72 hours, or at no less than ±0.02 min, applied to the midpoint initial calibration standard.

b. If the RTs for a continuing calibration verification standard fall outside the RT window, update the midpoint RT using that standard.

(1) Save this under the appropriate name to indicate an update has occurred.

(2) RTs cannot be updated more than once per day. All subsequent standards run within a 24−hour period must be within this window.

(3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Prepare a sequence of injections as suggested in the Calibration Section, along with the appropriate check standards.

2. Retention times of peaks in the samples are compared to the standard RT windows

   a. Peaks present on both columns that are also in the correct ratios to represent an aroclor are quantitated, and the high value is reported unless chromatographic anomalies are observed. See *T-PEST-WI9954*.

   b. Use a minimum of three to six peaks for quantitation, with the exception of certain mixes where it may be more accurate to use less peaks to avoid excessive overlap of patterns.

3. Continue running groups of samples/injections followed by check standards every 12 hours or 20 injections, whichever comes first.

   a. For projects where a known aroclor is present and at the request of clients, other aroclors can be run for the continuing check standard. For instance, a set of continuing standards may be AR484, AR164, IBLK.

   b. Aroclor 1262 and 1268 can be analyzed when requested. A full six point curve can also be run, depending on the project requirements.

4. If significant interference is present, schedule florisil and/or sulfuric acid cleanup. If elemental sulfur is present, TBA or copper the extract or have it put through GPC cleanup. If these techniques do not reduce the matrix problems, dilute the extract with hexane and adjust LOQs accordingly.

5. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

Calculations

1. The peak heights generated by the integration system are used to calculate the calibration factors (CF) for peaks of interest for each aroclor.

Usually, the six major peaks that are unique to each aroclor are chosen for quantitation, with the exception of 1221 where only three peaks are available.

2. Sample concentrations are calculated per peak using average calibration factor (AVG CF) from the initial calibration.

$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{FV}{IW} \times DF = \mu g/kg \text{ as received per peak}$$

Where:

FV = Final volume (mL)

IW = Initial weight (g)

DF = Dilution factor, as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

$$(\text{Result 1} + \text{Result 2} + \ldots + \text{Result } n) \;/\; n = \text{Average Result}$$

3. The surrogate results are determined using either AVGRF or linear curve:

Using AVGCF from the initial calibration:

$$\frac{\text{Sample Height}}{\text{AVCRF (CF)}} \times \frac{FV}{IW} \times DF = \mu g/kg \text{ as received}$$

b. Using linear curve from the initial calibration:

$$[(\text{Sample Height} - Y - \text{intercept})/\text{Slope}] \times \frac{FV}{IW} \times DF = \mu g/kg \text{ as received}$$

Where:

FV = Final volume

IW = Initial weight

DF = Dilution factor, as needed

4. See *T-PEST-WI9847* for all other equations related to the analysis.

Statistical Information/Method Performance

LCS, MS, and RPD are compared to the limits stored in the LIMS. The limits for LCS and MS are statistically derived. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Acceptance limits are generated according to *T-PEST-WI9980*. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to *QA-SOP11892* for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the manager. The department database is updated via a download from the LIMS.

When 8000D is referenced an annual lower limit of quantification (LLOQ) verification is required annually on at least one instrument. All instruments must have one at least every three years. LLOQV is required by 8000D.

Quality Assurance/Quality Control

QC Acceptance limits are established as statistical limits. See *T-PEST-WI9980* further information on monitoring and establishing limits.

A sodium sulfate blank and a sodium sulfate spike (LCS) are analyzed with every group of samples up to a maximum of 20. An MS/MSD is performed per batch. For wipes and when an MS/MSD cannot be performed, an LCSD will be extracted.

Aroclor 1016 and 1260 are routinely spiked; however, other aroclors can be spiked as requested by clients.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

See *T-PEST-WI10011* for details on QC acceptance criteria and corrective action.

Appendix I

PCT Analysis (Aroclor 5422, 5432, and 5460)

A. Standards:

1. PCT-Stocks- Aroclor 5432, 5442, and 5460 purchased as individually ampulated solutions from Accustandard at 35,000ug/L.

2. Surrogate Stock- Ultra ISM-320 containing TCX/DCB at 200,000ppb in acetone. Prepare an intermediate by diluting 0.25mL to 25mL of hexane.

3. MS stock of Aroclor 5442 - Absolute cat# 71791, 1000ug/L in Methanol.

4. Prepare working standards using the electronic standard database as a guide.

   a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

   b. The database contains the following information: solution description (ex. AR161), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

   c. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

d. The scheme for preparing the matrix and surrogate spiking solutions used in the extraction process are listed below.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
MS	Aroclor 5422 Stock	0.75	100	Acetone	Aroclor 5442 PCT Spike
SS	Pest Surr mix	1.5	1000	methanol	SW 846 surrogate

B. GC Chromatographic Analysis

The conditions listed serve as a guideline only and are typically the optimum operating conditions. The analyst may make any changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity, and/or improve separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

Detector: ECD

Detector temp: 330°C

Oven Temp: 110°C to 250° at 40°C/min, to 280°C at 20°C/min, to 330°C at 30°C/min, hold 9 min.

Carrier: Hydrogen at 3.6 mL/min

Makeup gas: N_2 at 60mL/min or equivalent

Injection size: 1-uL, direct injection

Injection temp: 225°C

C. Example of a Sequence:

1. Conditioner
2. IBLK
3. EVAL
4. AR161
5. AR162
6. AR163

7. AR164
8. AR165
9. AR166
10. AR481
11. AR482
12. AR483
13. AR484
14. AR485
15 AR486
16. AR541
17. AR542
18. AR543
19. AR544
20. AR545
21. AR546
22. PCT1
23. PCT2
24. PCT3
25. PCT4
26. PCT5
27. A4421
28. A4422
29. A4423
30. A4424
31. A4425
32. AR213
33. AR323
34. AR423
35. AR623
36. AR683
37. AR16xx
38. MDPCTX
39. MD16
40. IC16
41. IC48
42. IC54
43. Blank
44. LCS
45. 1234567
46. 1234567MS
47. 1234567MSD
48-62. Continue running samples
63. AR163
64. PCT3
65. A4423
66. IBLK

T-OE-PEST-WI10281 Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-OE-PEST-WI10922 Ultrasonic Extraction for PCBs in a Solid Matrix by Method 3550C
T-OE-PEST-WI10927 Microwave Extraction Method 3546 for PCBs in a Solid Matrix
T-PEST-WI10007 Preventative and Corrective GC Maintenance
T-PEST-WI10011 QC Data Acceptability and Corrective Action
T-PEST-WI10016 Setting Up Single Component Initial Calibrations
T-PEST-WI10022 Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PEST-WI9954 Interpretation of Chromatographic Data
T-PEST-WI9980 Monitoring QC Data Acceptance Limits

End of document

Version history

Version	Approval	Revision information
8	26.AUG.2016	
9	29.MAR.2018	

E-08

Analysis of Polychlorinated Biphenyls (PCBs) by 8082A in Aqueous Samples Using GC-ECD
(Rev. 6; 03/29/2018; T-PEST-WI9238)

LIMS ID
Analysis - 10591, 13092

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Revision Log
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Personnel Training and Qualifications
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Appendix I

Revision Log

Revision 6		**Effective Date:**	This version
Section	**Justification**	**Changes**	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Reference	Reflects current references	Added reference for 8000C and 8000D	
Cross Reference	References to higher level documents not necessary	Removed references 1-P-QM-QMA-9015390 and 1-P-QM-QMA-9017309	

Revision 6	**Effective Date:**	This version
Interference	Enhancement	Added interference from DDD, DDE, and DDT.
Scope	Reflects current LOQs	Updated PCTs from 0.5 ug/L to 1.0 ug/L
Definitions	Common terms defined in higher level documents	Removed section
Personnel Training and Qualifications	Reference to higher level documents unnecessary	Removed reference to 1-P-QM-QMA-9015390
Sample Collection, Preservation, and Handling	Reflects current extract storage conditions and preservation	Added extract storage condition. Removed information on preservation. Changed sample extraction hold date to none recommended.
Apparatus and Equipment	Enhancement	Reformatted section
Reagents and Standards	Reflects current standards	Removed individual 1260 standard. Updated Bottle Code 43 table Added the MS and SS working solutions are stored at ≤-10° C.
Extraction	Reflects current prep LIMS scans	Removed all prep LIMS scans except 11121 and 13093
Calibration	Reflects current practice	Removed wording on changing the septa prior to starting calibration Added AR16X to the sequence. Added step when new ICV does not meet criteria. Changed from using 5 levels to using 6 levels for the calibration. Changed from using level 3 for CCV to using level 4.
Calibration	Reflects 8000D	Added information on 8000D criteria. Added information that all individual peaks must meet the criteria.
Procedure	Reflects current practice	Removed GPC cleanup. Added information on raising reporting limits.
Calculations	Reflects current final and initial volumes	Added 2 ml and 250ml for final volume and initial volume, respectively
Statistical Information/ Method Performance	Reflects current process	Removed reference to 1-P-QM-QMA-9017309. Added wording similar to the SOP for SW 846 8082.
Statistical Information/ Method Performance	Reflects current method requirement	Added similar verbiage as the other department SOPS using 8000D.
Appendix 1	Reflects current PCT standards	Added manufacturer for individual standards. Updated table for MS Spike and surrogate spike. Added text indicating that the MS and SS working solutions are stored at ≤-10° C. Added 6 levels of PCBs.

Revision: 5		**Effective Date:** Nov 04, 2015
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Historical/ Local Document Number	LIMS scan relevant to this procedure	Add 13092 for PCTs
Sample Collection, Preservation, and Handling	Reflects current industry standard for refrigeration storage.	Changed to 0° to 6° C, not frozen.
Apparatus and Equipment	Reflects current instrumentation	Updated 7890 GC
Calibration	Reflects method criteria Enhancement Reflects current procedure	Changed wording to second column should meet 20% continuing calibration criteria. Added information on analyzing an EVAL to aid in identifying possible DDD, DDE, and DDT in the aroclor pattern. Added information on analyzing a single PCB 1016 standard to help with pattern recognition. Changed the RT window from 0.03 to 0.02 min.
Gas Chromatographic Conditions	Reflects current operating conditions	Updated GC conditions.
Calculation	Clarification	Added information on not using individual peaks where the value is <MDL in calculating the PCB concentration.
Appendix I	Clarification for PCTs	Removed table for calibration standard preparation and added information referencing the standard database. Updated GC conditions for analyzing PCTs.

Reference

1. T*est Methods for Evaluating Solid Waste*, SW-846, Method 8082A, February 2007.

2. Determinative Chromatographic Separations, SW-846, Method 8000D, July 2014

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #6654, 10241, 11112, 11113, 11114, 11116, 11117, 11118, 11119, 11120, 11121, 11123, 11126, 11960, 12026, 12822, 13086, 13093, 13183, 13187	Separatory Funnel Extraction by Method 3510C, 608 or 622 for Pesticides and PCBs in a Wastewater

Document	Document Title
T-OE-PEST-WI10281	Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-PEST-WI10011	QC Data Acceptability and Corrective Action
T-PEST-WI9954	Interpretation of Chromatographic Data
T-PEST-WI10007	Preventative and Corrective GC Maintenance
T-PEST-WI9980	Monitoring QC Data Acceptance Limits
T-PEST-WI10016	Setting Up Single Component Initial Calibrations
T-PEST-WI10022	Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses

Scope

This method is useful for identifying and quantitating the following polychlorinated biphenyls (PCBs) as Aroclors in aqueous matrices:

Compound	LOQ (µg/L)
Aroclor 1016	0.5
Aroclor 1221	0.5
Aroclor 1232	0.5
Aroclor 1242	0.5
Aroclor 1248	0.5
Aroclor 1254	0.5
Aroclor 1260	0.5

The following aroclors can also be analyzed for upon request by the client, usually on a project basis. Since aroclors 1262 and 1268 contain peaks that elute much later than 1260, the GC run time needs to be extended to ensure the entire pattern has eluted.

Compound	LOQ (µg/L)
Aroclor 1262	0.5
Aroclor 1268	0.5
Aroclor 5422	1.0
Aroclor 5432	1.0
Aroclor 5460	1.0

See Appendix I for GC operating conditions and calibration information when analyzing for Aroclor 5422, 5432, and/or 5460.

Limits of Quantitiation (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS.

Basic Principles

A portion of an aqueous sample is extracted serially with methylene chloride. The volume of sample extracted can be adjusted depending on the physical appearance of the sample and the amount sent for analysis. The extract is dried, concentrated, and exchanged into hexane. The PCBs are then identified and quantitated using gas chromatography (GC) with an electron capture detector (ECD). The extract may require further cleanup (by florisil, sulfuric acid, or copper) to reduce matrix interferences that introduce large, unresolvable peaks in the chromatogram (due to interferents such as phthalates, oxygenated organics, unsaturated organics, or elemental sulfur).

Reference Modifications

Gas Chromatography conditions are different than those listed in Method 8082A however, all QC criteria are met.

Interferences

A. Avoid contact with any plastic material during the extraction and analysis procedures to minimize interferences from phthalate esters.

B. Scrupulously clean all glassware to minimize interferences caused by laboratory contaminants.

C. An electron capture detector (ECD) is very sensitive to compounds that contain halogens and will also respond to many other compounds and materials including oxygenated organics, unsaturated organics, and elemental sulfur.

D. Extracts may require further cleanup if interferents are present. Refer to *T-OE-PEST-WI10281* for details on each cleanup procedure. Interfering materials can introduce large, unresolvable peaks into the chromatogram.

1. Use Florisil cleanup to reduce organics that can interfere (polar compounds).

2. Use GPC to remove sulfur and higher molecular weight organics.

3. Use copper or TBA cleanup to remove elemental sulfur.

E. Common single component pesicides such as DDT, DDD, and DDE may cause interference with the aroclor pattern. To ensure that the analyst is aware of this, a standard containing DDE/DDD/DDT will be run with each initial calibration.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

Gloves, lab coats, and safety glasses must be worn when preparing standards. Safety glasses must be worn around the GC where solvents and sample extracts are handled.

GC vials are disposed of in the designated lab container and subsequently lab packed for final disposal. All solvent waste is placed in designated containers in the laboratory then taken to the lab–wide facility by personnel trained in hazardous waste disposal.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs. .

Sample Collection, Preservation, and Handling

Samples are collected in amber glass containers with Teflon–lined caps and kept cool at 0° to 6°C, not frozen.

PCBs have no maximum recommended holding time for the extraction. Some agencies or projects may require a specific hold time for the extraction. If the hold time is specified in the project, this hold time must be followed. Sample analysis must be performed with 40 days of extraction. Extracts are stored in the freezer at ≤-10°C.

Apparatus and Equipment

1. HP 7890 gas chromatograph equipped with an electron capture detector, or equivalent

2. Columns:

   a. Phenomonex MultiRes I – 30 m × 0.32 mm × 0.5 µm, or equivalent

   b. Phenomonex MultiRes II – 30 m × 0.32 mm × 0.25 µm, or equivalent

3. Integrating system such as Chrom Perfect® by Justice Laboratory Software, or equivalent. Chrom Perfect® is a data system capable of storing and reintegrating chromatographic data and determining peak areas using a forced baseline, area summation, baseline projection, and performing baseline compensation as required.

4. Various sizes of Class A volumetric flasks, pipettes, and syringes

Reagents and Standards

A. Reagents

   1. Follow manufacturer's guidelines for storage conditions.

   2. Hexane for autosampler rinse vials. Stored at room temperature up to 1 year.

   3. UPC (Ultra Pure Carrier) helium for carrier gas.

   4. UPC nitrogen for detector make-up gas.

5. UPC hydrogen carrier gas, either bottled or from a generator.

B. Standards

1. All standards are prepared using Class A volumetric pipettes, syringes, and flasks.

2. All weights are made on an analytical balance.

3. Unopened ampules are stored according to the manufacturer's instructions and are stable until the expiration date provided by the manufacturer.

4. All prepared standard solutions are stored at ≤-10° C in labeled containers for up to 6 months.

If comparison with check standards indicates a problem prior to expiration date, the standards must be replaced.

5. Aroclor 1016 stock – Restek 32006 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane.

6. Aroclor 1016/1260 stock – Restek 32039 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane.

7. Aroclor 1221 stock – Restek 32007 at 100,000 ppb in hexane. Two separate lots are kept on hand to provide a second source.

8. Aroclor 1232 stock – Restek 32008 at 100,000 ppb in hexane. Two separate lots are kept on hand to provide a second source.

9. Aroclor 1242 stock – Restek 32009 at 100,000 ppb in hexane. Two separate lots are kept on hand to provide a second source.

10. Aroclor 1248 stock – Restek 32010 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane. Two separate lots are kept on hand to provide a second source.

11. Aroclor 1254 stock – Restek 32011 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 to 10 mL of hexane. Two separate lots are kept on hand to provide a second source.

12. Initial calibration Verification standard (ICV) Stocks:

a. Aroclor 1016 - Accustandard cat # C-216S-H-10X at 1,000,000 ppb

Aroclor 1260 Accustandard Cat# C-260S-H-10x at 1,000,000 ppb

Prepare an intermediate by diluting 1mL of each to 10 mL of hexane.

b. Aroclor 1248 - Accustandard cat# C-248S-H-10X at 1,000,000 ppb.

Prepare an intermediate by diluting 0.5 mL to 5 mL of hexane

c. Aroclor 1254 – Accustandard cat#C-254S-H-10X at 1,000,000 ppb.

Prepare an intermediate by diluting 0.5 mL to 5mL of hexane.

13. Surrogate stock (SS) – Supelco 861284 containing DCB/TCX at 200,000 ppb in acetone.

14. 1016/1260 Matrix spike (MS) stock: Restek 32039. Each at 1,000,000 ppb in hexane. Also used as a second source stock. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

15. Aroclor 1262 stock – Restek #32409 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

16. Aroclor 1268 stock – Restek #32410 at 1,000,000 ppb in hexane. Prepare an intermediate by diluting 1 mL to 10 mL of hexane.

17. Instrument Blank (IBLK) – Surrogate Stock (SS) – Supelco #861284 containing Decachlorobiphenyl (DCB) and Tetrachlorometaxylene (TCX) at 200,000 ppb each in acetone.

18. Prepare working standards using the electronic standard database as a guide.

a. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

b. The database contains the following information: solution description (ex. AR161), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

c. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

d. For Bottle Code 43's

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
PCB Spike	1016/1260 MS Stock	1.25	250	Acetone or methanol	Water Spike
SS	SS Stock	3	2000	Acetone or methanol	SW-846 Water surrogate – identical to that prepared for Pest/PCB analyses

e. For Bottle Code 153's

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
Mini Sep. PCB Spike	PCB Spike	12.5	50	Acetone or methanol	Water Spike
SS	SS Stock	0.375	1000	Acetone or methanol	SW-846 Water surrogate – identical to that prepared for Pest/PCB analyses

The MS and SS working solutions are stored $\leq$-10° C

19. An initial calibration verification standard must also be prepared at a concentration at the mid-point of the calibration for 1016, 1260, 1248, 1254 using a stock solution purchased from a different vendor or different lot than that used for the calibration standards.

20. Additional standards and preparations are listed in Appendix I for Aroclor 5442, 5432, and 5460 (PCT analysis).

Extraction
See organic extraction Analysis # 11121 and 13093.

Gas Chromatographic Conditions
The conditions listed are usually the optimum operating conditions but can vary to improve the sensitivity, linearity, and overall chromatography or shorten run times on each GC system.

Detector	ECD
Detector Temperature	330°C
Oven Temperature	110°C 40°C/min. to 250°C 20°C/min. to 280°C 30°C/min to 330°C Hold until DCB elutes ~ 2 min.
Column A	MR I
Column B	MR II
Carrier	Hydrogen at 12 psi, 5ml/min constant flow. (Can be substituted with Helium)
Makeup gas	N_2 at 80 mL/min. for Agilent GCs
Injection size	1 µL, direct injection
Injection Temperature	225°C

A Merlin microseal can be used in place of traditional septum.

Calibration

A. Fill the autosampler rinse vials with clean solvent or replace vials that appear dirty.

B. Prepare a sequence using the following order of injections:

1. Conditioner
2. IBLK
3. EVAL
4. AR161
5. AR162
6. AR163
7. AR164
8. AR165
9. AR166
10. AR481
11. AR482
12. AR483
13. AR484
14. AR485
15. AR486
16. AR541
17. AR542
18. AR543
19. AR544
20. AR545

21. AR546
22. AR214
23. AR324
24. AR424
25. AR624
26. AR684
27 AR16X
28. MD16 (MDL)
29. IC16X (ICV)
30. IC48X (ICV)
31. IC54X (ICV)
32. Blank
33. LCS
34. 1234567
35. 1234567ms
36. 1234567msd
37.– 47. Continue running samples
48. AR164 (CCV)
49. IBLK

1. Note for our routine PCBs: The AR21, AR32, and AR42 are level 4s in the ICAL and are entered as AR214 and AR424 in the ICAL. Also the AR62 and AR68 are both level 4s and are listed as AR624 and AR684 in the sequences.

2. A single point of Aroclor 1016 is analyzed with the calibration to aid in better pattern recognition in the samples. Aroclor 1016 and 1242 have similar patterns with the exception of the smaller trailing peaks at the end of the Aroclor 1242 pattern. This is identified in the sequence as AR16X.

3. A DDT/Endrin breakdown standard is run with each ICAL in order to provide the retention times of p,p-DDE, p,p-DDD, and p,p-DDT which may interfere with the pattern for aroclor 1254, 1248, and 1260.

4. See Appendix I for a sequence example when the analysis of PCTs (Aroclors 5442, 5432, and 5460) is requested.

D. Continue running groups of samples/injections. Each bracket must contain no more than 20 samples/injections and last no more than 12 hours for Method 8082A. Each bracket is followed by the continuing calibration check standard and an instrument blank (IBLK).

E. The conditioner injection is usually a standard or sample that has already been injected.

1. The conditioner is used to prime the system.

2. It is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

F. Hexane blanks can also be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system.

G. Instrument blanks (IBLK) may also be run with the continuing calibration standards. This is optional but frequently requested for projects.

1. The instrument blank (IBLK) is injected after the conditioners but before the initial calibration.

2. It is used to determine that the instrument is free of background noise or contamination.

H. For projects where a known aroclor is present and at the request of clients other aroclors can be run for the continuing check standard. For instance, a set of continuing standards can be AR484, AR164, and IBLK.

I. Aroclor 1262 and 1268 can be analyzed when requested. A full six point curve can also be run, depending on the project requirements.

J. Initial Calibration (ICAL)

1. Calibrate for the aroclors using the six levels of 1016, 1260, 1248, and 1254. Use the single point for 1221, 1232, 1242, 1262, and 1268 when needed. As an option, 1248 and 1254 can use a single point calibration.

2 An external standard calibration based on the average calibration factor (AVG CF) for all analytes is used for quantitation where the %RSD is ≤20%.

3. The surrogate standards are calibrated using the AR16 levels using AVGCF unless the % RSD is >20%.

4. If the RSD is > 20%, use a calibration curve.

a. Use a linear fit.

b. The correlation coefficient must be >0.99 to be a valid fit.

c. Extrapolate or force to zero is not allowed. Set the zero to ignore. See *T-PEST-WI10016* for more details.

When 8000D is referenced and curve fits are used in the initial calibration, percent error (%E) must be calculated for each calibration level. Standard levels at or below the LOQ must meet +/

- 50%, standards above the LOQ must meet +/-30%. (%error = (true concentration - calculated concentration)/true concentration)*100). All individual peaks used to calculate the aroclor pattern must meet the %E criteria.

5. If the 0.99 curve coefficient cannot be met:

a. Inspect the data points to see if one or more calibration level became concentrated due to solvent evaporation or degraded over time.

b. Reinject or remake the standard if a specific calibration level has concentrated due to solvent evaporation, or degraded over time..

c. Perform instrument maintenance as needed. See *T-PEST-WI10007* for troubleshooting linearity problems.

6. The calibration range for the aroclors is 50ug/L to 1000 µg/L. Level 1 is equivalent to the Limit of Quantitation.

7 Set up the aroclor calibration data in a custom program under Datalog.

The retention times of the peaks to use for identifying and quantifying the aroclors are entered into the calibration file along with the corresponding peak heights and concentrations. See *T-PEST-WI10022* for details.

8. Ensure the peaks in the standards are labeled properly, including the surrogates in all injections that contain them.

9. Set the scaling of chromatograms and peak integration parameters so that the size of the peaks for each compound of interest are approximately 2 to 3 mm in height at the concentration of the method detection limit (MDL).

a. Ensure all peaks in the MDL standard are integrated.

b. By running the 1016/1260 MDL standard, the majority of peaks of all aroclors are represented.

K. Initial Calibration Verification (ICV)

1. Verify the calibration curves using the ICV mixtures injected directly after the full ICAL.

2. The % difference of the concentrations for these must be within 20% difference of the nominal concentration. All the individual peaks used in the ICAL for the PCB pattern must meet this criteria.

a. If this criteria is not met, reinject the ICV.

b. If the criteria is not met again, then prepare a new standard.

c. If the criteria is not met after analyzing the new ICV standard, then a recalibration is performed.

L. Continuing Calibration Verification (CCV)

1. Calibration verification is performed after each set of twenty injections (samples, QC, blanks, etc.) or 12 hours, whichever comes first.

2. Use AR16 to evaluate the calibration of the aroclors (or other aroclors as requested for particular clients or projects).

3. Other aroclors can also be used along with AR16. This must be done if a site has prior history of containing a specific aroclor, or as requested for client projects or to meet other regulatory requirements.

4. The concentration quantitated for the continuing calibration check standards must be within 20% difference (%D) of the nominal concentration for each compound. All the individual peaks used in the ICAL for the PCB pattern must meet this criteria.

5. Samples must be bracketed with compliant standards.

a. Exception: If the standard following a sample is outside the ±20% but exhibits increasing response, the samples before it do not have to be reinjected if the target analytes are not detected. A comment must be added to the analytical report.

b. If two consecutive/sequential CCVs fail, corrective action must be taken, which can include performing injection port maintenance, baking the instrument or other types of maintenance. Two consecutive/sequential CCVs must meet the criteria or an initial calibration must be performed before sample analysis can continue.

c. If confirmation of target analytes is needed, then the second column should meet the 20% continuing calibration criteria, as well as all initial calibration criteria. if either of these are not met, a comment must be added to the analytical report.

6. The instrument blank (IBLK) is injected after each set of continuing calibration verifications when requested.

a. It must be evaluated as a water matrix against the water MDL/LOQs.

b. The IBLK must not have any target compounds above the reporting limits.

(1) If a target analyte is detected in the IBLK, any associated samples with a detection for that same target must be evaluated.

(2) Unless the concentration in the sample is more than 10x the IBLK value, the sample must be reinjected after another compliant IBLK.

(3) Instrument maintenance, like baking the system or injection port maintenance is usually necessary to clean up instrument.

7. Retention time (RT) windows

a. Established as 3× the standard deviation determined over 72 hours or at no less than ±0.02 min, applied to the mid−point initial calibration standard.

b. If the RTs for a CCV fall outside the RT window, update the mid−point RT using that standard.

(1) Save this under the appropriate name to indicate an update has occurred.

(2) RTs cannot be updated more than once per day. All subsequent standards run within a 24−hour period must be within this window.

(3) If RTs are not consistent, the cause must be investigated and corrective action taken.

Procedure

1. Make injections via an auto sampler.

2. Samples are analyzed according to the sequence in the calibration section above, along with the appropriate check standards.

3. Retention times of peaks in the samples are compared to the standard RT windows.

Peaks present on both columns that are also in the correct ratios to represent an aroclor are quantitated and the high value is reported unless chromatographic anomalies are observed. See *T-PEST-WI9954*.

4. Use a minimum of three to six peaks for quantitation, with the exception of certain mixes where it can be more accurate to use less peaks to avoid excessive overlap of patterns.

5. If significant interference is present, schedule florisil and/or sulfuric acid cleanup. If elemental sulfur is present, copper treat the extract. If these techniques do not reduce the matrix problems, dilute the extract with hexane, analyze, and adjust the LOQs accordingly. Also, the reporting limit for individual aroclors may be raised in instances where matrix interference hinders the recognition of an aroclor pattern in the specified retention time window. The reporting limit is calculated by averaging the concentration of at least 3 peaks in the pattern.

6. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

Calculations

A. See *T-PEST-WI9847* for details on all calculations/equations used to evaluate the initial and continuing calibration.

B. Calculation of results is performed according to the following procedures:

1. The peak heights generated by the integration system are used to calculate the calibration factors (CF) for peaks of interest for each aroclor. Usually, the six major peaks that are unique to each aroclor are chosen for quantitation with the exception of 1221 where only three peaks are available.

2. Sample concentrations are calculated per peak using average calibration factor (AVG CF) from the initial calibration for 1016, 1260, 1248, and 1254 where a multi-point calibration is run, single point CF for 1221, 1232, 1242, (a multi−point calibration can also be run for these as requested or necessary to meet client or regulatory agency requirements). Do not use individual peaks that have values <MDL in quantitation.

3.

$$\frac{\text{Sample Height}}{\text{AVG CF (CF)}} \times \frac{FV}{IV} \times DF = \mu g/L \text{ as received}$$

Where:

FV = Final volume – 2 or 10 mL

IV = Initial volume – 250 or 1000 mL

DF = Dilution factor, as needed

The final result that is reported is determined as the average of the result for each peak chosen for quantitation:

4. The surrogate results are determined using either AVGRF or linear curve:

$$(Result\ 1 + Result\ 2 + \ldots + Result\ n) / n = Average\ Result$$

a. Using AVGCF from the initial calibration:

$$\frac{Sample\ Height}{AVG\ CF\ (CF)} \times \frac{FV}{IV} \times DF = \mu g/L\ as\ received$$

b. Using linear curve from the initial calibration:

$$[(Sample\ Height - Y - intercept)/Slope] \times \frac{FV}{IV} \times DF = \mu g/L\ as\ received$$

Where:

FV = Final volume – 2 or 10 mL

IV = Initial volume – 250 or 1000 mL

DF = Dilution factor, as needed

Statistical Information/Method Performance

LCS, MS, and RPD are compared to the limits stored in the LIMS. The limits for LCS and MS are statistically derived. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Acceptance limits are generated according to *T-PEST-WI9980*. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to *QA-SOP11892* for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the manager. The department database is updated via a download from the LIMS.

When 8000D is referenced an annual lower limit of quantification (LLOQ) verification is required annually on at least one instrument. All instruments must have one at least every three years.

Quality Assurance/Quality Control

A reagent water blank and a reagent water spike (LCS) are analyzed each day with every batch of samples (batch size is 20). An MS/MSD is performed per batch as long as there is ample volume of a sample in the batch. If an MS/MSD cannot be performed, an LCSD must be extracted.

Aroclor 1016 and 1260 are routinely spiked, however, other aroclors can be spiked as requested by clients.

DCB and TCX are added as surrogates to each sample and QC to monitor the efficiency of the extraction, the operation of the autosampler, and to monitor retention times throughout the GC run.

If any client, agency, or state has more stringent QC or batch requirements, these must be followed.

See *T-PEST-WI10011* for details on QC acceptance criteria and corrective action.

Appendix I

PCT Analysis (Aroclor 5442, 5432, and 5460)

A. Standards:

PCT-Stocks - Aroclor 5432 (Accustandard T-4325), 5442, and 5460(Accustandard T-460S) purchased as individually ampulated solutions from Accustandard at 35,000ug/L.

MS stock of Aroclor 5442 - Absolute Cat. #71791. 1000ug/mL in Methanol.

Prepare working standards using the electronic standard database as a guide.

1. In the database, choose the category (i.e. working spike, surrogate, intermediate, etc) and the required standard.

2. The database contains the following information: solution description (ex. AR161), parent solution name, aliquot used, final volume, solvent used, concentration of each compound in the solution, and expiration date. The working standards have an expiration date of 6 months.

3. The calibration scheme begins at or near the reporting limit through a 20 fold of the initial calibration level.

4. The scheme for preparing the matrix and surrogate spiking solutions used in the extraction process are listed below. The MS and SS working solutions are stored at ≤-10° C.

Standard Name	Parent Solution	Aliquot (mL)	Final Volume (mL)	Solvent	Description
MS	5422 MS Stock	0.625	125	Acetone or methanol	PCT Water Spike
SS	SS Stock	3	2000	Acetone or methanol	PCB surrogate

B. GC Chromatographic Conditions

Detector: ECD
Detector temp: 300°C
Oven Temp: 110°C to 250° at 40°C/min, 280 to 330°C at 30°C/min, hold 9 min.
Carrier: Hydrogen at 5.0 mL/min
Makeup gas: N_2 at 80mL/min or equivalent
Injection size: 1-uL, direct injection
Injection temp: 225°C

The conditions listed serve as a guideline only and are typically the optimum operating conditions. The analyst may make any changes to the chromatographic conditions to improve the speed of analysis, linearity, sensitivity, and/or improve separation if initial and continuing calibration criteria and quality assurance criteria listed within this analysis document are met.

C. Sequence

1. Conditioner
2. IBLK
3. EVAL
4. AR161
5. AR162
6. AR163
7. AR164
8. AR165
9. AR166
10. AR481
11. AR482

12. AR483
13. AR484
14. AR485
15. AR486
16. AR541
17. AR542
18 AR543
19. AR544
20. AR545
21. AR546
22. PCT1
23. PCT2
24. PCT3
25. PCT4
26. PCT5
27. A4421
28. A4422
29. A4423
30. A4424
31. A4425
32. AR214
33. AR324
34. AR423
35. AR624
36. AR684
37. MD16
38 MDPCTX
39. IC16
40. IC48
41. IC54
42. Blank
43. LCS
44. 1234567
45. 1234567MS
46. 1234567MSD
47-61. Continue running samples
62. AR164
63. PCT3
64. A4424
65. IBLK

T-OE-PEST-WI10281 Cleanup Procedures for the Extraction of Pesticides and Polychlorinated Biphenyls (PCBs)
T-OE-PEST-WI10920 Separatory Funnel Extraction by Method 3510C, 608 or 622 for Pesticides and PCBs in a Wastewater
T-PEST-WI10007 Preventative and Corrective GC Maintenance
T-PEST-WI10011 QC Data Acceptability and Corrective Action
T-PEST-WI10016 Setting Up Single Component Initial Calibrations

T-PEST-WI10022 Using "Datalog" Software for Data Acquisition of Multicomponent Pesticides/PCBs
T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PEST-WI9954 Interpretation of Chromatographic Data
T-PEST-WI9980 Monitoring QC Data Acceptance Limits

End of document

Version history

Version	Approval	Revision information
5	04.NOV.2015	
6	29.MAR.2018	

E-09

% Moisture Calculation
(Gravimetric) by SM 2540
G-1997
(Rev. 16; 12/28/2017; S-SS-
WI10697)

LIMS ID
0111, 6111, 7611, 11624, 12845

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Revision Log
Reference
Cross Reference
Scope
Basic Principles
Reference Modifications
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control
Comments

Revision Log

Revision 16		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Title	Enhancement	Added method reference
Cross Reference and Throughout document	Clarification	Removed the level 2 documents that weren't necessary

Revision 16	**Effective Date:** This version	
Apparatus and Equipment	Enhancement	Added Tongue Blades or equivalent and pipette
Procedure 1.,11.	Clarification	Changed text to reflect calibration check
Procedure 6.	Reflects current practice	Added instruction on sampling sludges with a high liquid content

Revision: 15	**Effective Date:** JUL 14, 2016	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Reference	State requirement	Added 2540 G 18th edition reference
Scope	State requirement	Added the state specific requirement for 2540 G 18th edition

Reference

1. SM 2540 G-1997 % Moisture Calculation - Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 2540 G (modified).

2. Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass, ASTM D 2216-98.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
S-SS-WI10696	Maintenance of Desiccators
QA-SOP11880	Balance, Syringe, Pipette Verification
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is for the determination of moisture in most solid and semisolid samples.

1. SM 2540G-1997 %Moisture- Dry Wt Calculation is the preferred method when performing the moisture procedure and is used as our default method.

2. Analysis 6111 is used for Client Account #11187 only. ASTM D 2216-98 is only used for analysis 6111.

3. Analysis 7611 is used for sieved samples only. All other samples are analyzed by analysis 0111.

4. Analysis 11624 is used for Client Account #13013 only. The procedure follows %Moisture-Dry Wt Calculation but has client specific QC criteria.

5. Analysis 12845 is used for Client Account #12528 only. The procedure has specific QC and LCS requirements.

Basic Principles

A measured amount of sample is placed in an empty tared pan and dried in an oven at 103° to 105°C. The decrease in weight of the sample, after drying, is calculated as the moisture content of the sample.

Reference Modifications

The method has been modified by using purchased aluminum foil pans that are not preconditioned, as this step is unnecessary.

SM 2540G-1997 %Moisture- Dry Wt Calculation requires multiple weights to be taken until the change in weight from the previous weight is within 4% or 50 mg (whichever is less). The modification is based on a study that was performed which determined that a constant weight was achieved after the initial drying cycle and therefore multiple weights were not necessary.

Interferences

The determination of moisture in solid and semisolid materials is subject to error due to loss of ammonium carbonate and volatile organic matter during drying.

Non−representative, nonhomogeneous materials, such as stones, are excluded from the analysis if their inclusion is not desired in the final result.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local state laws and regulations.

See Chemical Hygiene Plan for general information regarding employee safety, waste management and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing the analysis must work with an experienced technician for a period of time until they can independently perform the analysis. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control samples (or alternatively, one blind sample for the DOC) that are carried through all steps of the analysis and meet the defined acceptance criteria for the LCS.

Sample Collection, Preservation, and Handling

Samples for moisture analysis must be kept in an unpreserved container and refrigerated at 0 to 6°C, not frozen.

Apparatus and Equipment

1. Disposable aluminum foil pans, or equivalent
2. Oven maintained at 103° to 105°C. Adjust as needed to stay in this range.
3. Analytical balance, or equivalent.
4. Desiccators, or equivalent.
5. 6 X 3/4 Tongue Blades or equivalent

6. 1/10 inch opening pipette

7. The LLENS is computer program that integrates a PC with an analytical balance to collect data directly from the balance. It also organizes the data, performs calculations and transmits the final results to the central computer system. The LLENS system must be used for moisture analysis. .

Reagents and Standards
Alternate weights are acceptable but final concentrations must remain the same.

Standard solution of 10.5% NaCl: Add 895 ± 0.5g reagent water to 105 ± 0.2g NaCl in a beaker and mix thoroughly. Store in a 1000 mL clear glass bottle at 0 - 6°C, not frozen. This solution is used as prepared. Stable 6 months. (Concentration of solution is approximately 89.5% moisture.)

Calibration
Refer to *QA-SOP11880* for calibration procedures for analytical balances.

Procedure
1. Verify that the balance calibration check has been performed each day before use.

2. Set up the batch in LLENS.

3. Number (for identification) the purchased disposable aluminum foil pans before use.

4. Place one aluminum foil pan on a zeroed balance. Record pan tare weight in LLENS.

5. With the aluminum foil pan remaining on the balance, again zero the balance.

6. Place 5 to 10 grams of well−mixed sample into the pan.

NOTE: Most samples allow the use of a tongue blade for sampling but a pipette with a 1/10 inch opening must be used when sampling liquid sludge's or any matrix with significant amounts of liquid. These sample types must be shaken vigorously before sampling. The sample aliquot must be taken from the middle of the sample directly after shaking the sample.

7. Record the weight of the sample in LLENS

8. Record the date and time on the LLENS batch worksheet and then place the pans containing sample into the oven.

   a. Dry at 103° to 105°C for 8 to 48 hours.

   b. Oven temperatures are monitored and logged by the ETM (Environmental Temperature Monitoring) System through the use of a stationary probe. These temperatures are recorded and maintained within the ETM software.

9. Record the date and time on the LLENS batch worksheet, and then remove the pans containing dried sample from the oven.

10. Cool in a desiccator for 1 hour.

NOTE: Check desiccant to verify that the indicator crystals are still blue and not pink. SOP *S-SS-WI10696*, Maintenance of Dessicators, describes this procedure.

11. Again, verify that the balance calibration check has been performed each day before use.

12. Use tongs to place the pan containing dried sample on a zeroed balance. Record weight of pan and dried sample in LLENS.

Calculations

$$\%moisture = \frac{A - B}{C} \times 100$$

Where:

A = Weight of sample and container before drying (in grams)

B = Weight of sample and container after drying (in grams)

C = Weight of sample before drying (in grams)

Statistical Information/Method Performance

Refer to *QA-SOP11892*

Quality Assurance/Quality Control

Calibration verification of the balances must be performed each day before use.

An 89.5 % LCS must be run on each batch. Batch size is limited to 20 samples or less.

When sample volume permits, a sample matrix duplicate must be run with each group of not more than ten samples unless a client specifies otherwise.

See LIMS for current quality control (QC) acceptance windows.

Comments

1. The analysis report for 6111 must include a comment stating, “A 5 g aliquot was dried at 103°-105°C and calculated as %moisture.”

2. When a client submits a designated sample for QC, the matrix spike (MS) sample is entered for scan 0118.

   a. This allows the LLENS program to upload the moisture value from the client−designated background sample; this moisture value is often needed in the calculations of other analyses.

   b. A client−submitted continuation sample is also entered for scan 0118 for the same reason.

   c. Scan 6118 is specific to moistures done for Client Account #11187 only.

   d. Scan 7618 is specific to moistures done on sieved samples only.

   e. Scan 11625 is specific to moistures done for Client Account #13013 only.

   f. Scan 12846 is specific to moistures done for Client Account #12528 only.

   g. All other accounts use scan 0118.

3. When a client submits a designated sample for QC, the matrix spike duplicate (MSD) sample is entered for scan 0118 and scan 0121. (Scan 0118 provides the result for the MS and scan 0121 for the sample matrix duplicate.)

   a. Scan 6121 is specific to moistures done for Client Account #11187 only.

   b. Scan 7621 is specific to moistures done on sieved samples only.

   c. Scan 11626 is specific to moistures done for Client Account #13013 only.

d. Scan 12847 is specific to moistures done for Client Account #12528 only.

e. All other accounts use scan 0121.

QA-SOP11880 Balance, Syringe, Pipette, and Labware Verification
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
S-SS-WI10696 Maintenance of Dessicators

End of document

Version history

Version	Approval	Revision information
15	14.JUL.2016	
16	14.DEC.2017	

E-10

Total Suspended Solids (TSS) - Gravimetric by SM 2540 D2011 or SM 2540 D1997 and Total Filtered: Total Volume Test by NJDEP in Waters (Rev. 11; 12/24/2018; T-WC-WI11604)

LIMS ID
Analysis 10457, 13858, 14115

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Revision Log

Revision:	11	**Effective Date:**	This version
Section	**Justification**		**Changes**
Revision Log	Formatting requirement per 1–P–QM-QMA-9017356		Removed revision logs up to the previous version
Document Title	Addition		Added SM 2540 D-2011
Throughout document	Update to D4		Changed Cross References 2-5 to D4 document numbers
Reference 1.	Correction		Added SM 2540 D-1997
Basic Principles	Clarification		Rewritten to explain drying/weighing cycle. Added "weight change is less than 4% of the previous weight or ≤ 0.0005g, whichever is less."
Interferences	Clarification		Included how samples over the residue limit are treated
	Requirement		Added reagent grade and storage container.

Revision:	11	**Effective Date:**	This version
Reagents and Standards A.			
Procedure 8	Clarification		Changed name of step from analysis to filtration
Procedure A.8.b,e,f	New process		In addition to the volume used, the initial and all subsequent volumes of the filtering process will be recorded.
Procedure A.8.e-g	New process		Reorganized steps to match current process
Procedure A.8.h	Reflects current process		Removed original step A.8.h. Renumbered step 9 as step A.8.h
Procedure A.8.h, i (previously A.9,10)	This is part of the filtration process		Renumbered A.9,10 as step A.8.h and i. Renumbered following steps accordingly.
Procedure A.9.	New process		Added instructions for calculating the sample volume filtered.
Procedure A.12, 14 (previously (A.14,16)	Clarification		Distinguished between the two analyses drying requirements.
Procedure A.13 (previously A.15)	Reflects method reference		Changed cooling time to "cool to balance temperature".
Attachment I	Addition		Added comment #9388

Revision:	10	**Effective Date:**	09/28/2016
Section	**Justification**		**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356		Removed revision logs up to the previous version
Document Title	Enhancement		Add acronym TSS and rearranged wording
Cross Reference	Higher level documents are not required to be referenced Unnecessary references for this procedure		Removed reference to 1-P-QM-QMA-9015390, 1-P-QM-QMA-9017328, 1-P-QM-QMA-9021767, 1-P-QM-QMA-9015389 1-P-QM-PRO-9015422, 1-P-QM-PRO-9015424
Basic Principles	New process Correction		Noted Attachment I is a sieving prescreen Information on drying times moved out of the scope and into this section
Apparatus and Equipment	New process		Added items needed for Attachment I prescreen
Procedure	New process		Added sentence to see Attachment I for samples requiring prescreening
Quality Assurance/ Quality Control	Reflects current procedure		Added that the duplicate determinations should agree within 5% of their average weight
Attachment I	New process		Added Attachment I

Reference

1. *Standard Methods for the Examination of Water and Wastewater*, Method 2540 D-2011.

2. *Standard Methods for the Examination of Water and Wastewater*, Method 2540 D-1997.

3. NJDEP

4. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #0207, 0208	Fixed Suspended Solids (Gravimetric) (#207) Volatile Suspended Solids (Gravimetric) (#208) by SM 2540 E - 1997
T-WC-WI10359	Instructions for Collecting Data on the LLENS System
T-WC-WI10360	Quality Control Data for Wet Chemistry
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits

Scope

Analysis #10457 and Analysis #13858 are used for the determination of total suspended solids (TSS) in potable, surface and saline water, domestic and industrial wastewater in a range up to 20,000 mg/L. The reporting limit for this analysis varies with the sample volume. LIMS may round the reporting limit during the calculation routine.

Analysis #14115 for the Total Filtered:Total Volume test determines the ratio of filtrate to total volume.

Basic Principles

For Analysis #10457 and Analysis #13858 a well–mixed sample is filtered through a glass fiber filter and the residue on the filter is dried in an oven at 103° to 105°C. The increase in weight of the filter represents the TSS.

Analysis #10457 requires repeat drying, cooling, desiccating and weighing cycles until the weight change is less than 4% of the previous weight or ≤ 0.0005g, whichever is less.

Analysis #13858 records only one weight after a 16 hour drying cycle.

The appropriate analysis scan is typically determined by the sample state of origin.

For Analysis #14115, a 1–liter sample is filtered for up to 6 hours. The filtrate is measured and the ratio of filtrate to total volume is calculated.

Upon client request, a sieving prescreen which is outlined in Attachment I can be performed but a comment must be added to the analytical report that states that the sample was sieved prior to the TSS analysis.

Interferences

Samples high in dissolved solids may yield positive interferences. If the sample is over the residue limit of 0.2 g, the sample is reanalyzed at a reduced volume.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples must be unpreserved, collected in glass or plastic containers and refrigerated at 0° to 6°C, not frozen.

Analyze samples for Analysis #10457 and Analysis #13858 within 7 days of collection to minimize microbiological decomposition of the solids.

The holding time for Analysis #14115 is not specified.

Exclude non-representative, nonhomogeneous materials, such as leaves and sticks, from the analysis if their inclusion is not desired in the final result.

Apparatus and Equipment

A. For Analysis #10457 and Analysis #13858

1. ProWeigh Filters from Environmental Express – pre-weighed 47 mm glass fiber filters

2. ProWeigh Filters from Environmental Express – pre-weighed **volatile** 47 mm glass fiber filters or equivalent

3. Filtration apparatus

4. Desiccators.

5. Oven maintained at 104° ± 1°C. Adjust as needed to stay in this range.

6. Analytical balance.

7. LLENS - computer program that integrates a PC with an analytical balance to collect data directly from the balance. It also organizes the data, performs calculations and transmits the final results to LIMS. LLENS must be used for TSS whenever possible to facilitate the transfer of the data (see *T-WC-WI10359* for instructions).

B. For Analysis #14115

1. Filtration apparatus (2-4 per sample)

2. 0.45um filters

C. For Pre-screen sieving

1. Standard sieves with ½" and ¼" openings

2. 3-gallon bucket

Reagents and Standards

Alternate weights and volumes are acceptable as long as final concentrations remain the same.

A. TSS Working Standard (150 mg/L)

1. Dry 1,8 Naphthalimide, 98%, for approximately 1 hour at 104° ± 1°C.

2. Desiccate approximately 1 hour.

3. Dissolve 0.1500 ± 0.005 g Naphthalimide in approximately 700–mL of reagent water in a 1000–mL volumetric flask.

4. Mix very well.

5. Dilute to 1000 mL with reagent water.

6. Store at room temperature.

7. Stable 30 days.

8. Prior to use, Naphthalimide can be weighed out and stored in small glass vials at room temperature.

Calibration

A balance calibration check and leveling check must be performed each day before use.

Procedure

NOTE: Upon client request, a sieving prescreen which is outlined in Attachment I can be performed. After the completion of that process, continue with Procedure A.

A. Analysis #10457 and Analysis #13858

1. Batch samples for analysis in the LLENS system.

2. Assemble filtration apparatus.

3. Assign a pre-washed, pre-weighed filter to each client sample and each QC sample on the batch. If fixed or volatile suspended solids are to be performed use a pre-weighed **volatile** filter and follow instructions in Analysis #0207, 0208.

4. Remove the assigned filter from its aluminum carrying dish and place on the filtration unit.

5. Apply vacuum to the filtration apparatus.

6. Wet filter with small amount of reagent water to seat it.

7. Quality Control samples preparation

   a. Analyze the method blank by using 1000 mL of reagent water.

   b. Analyze the LCS/LCSD using 1000 mL of the 150 mg/L TSS working standard for each QC standard.

   c. Prepare duplicates by analyzing equal portions of sample.

8. Sample filtration

   a. Shake sample vigorously.

   NOTE: If the sample does not maintain a homogeneous state until the aliquot is taken, then magnetically stir the sample while pipetting the appropriate aliquot onto the filter.

b. Using a graduated cylinder or wide mouth pipette, measure 1000 mL of sample or a smaller portion if the sample matrix dictates. Record this volume in the 'initial volume' column.

c. Rapidly transfer the aliquot to the filtration unit.

d. Still under vacuum, filter the sample.

e. Repeat steps 8b-d until an appropriate total volume for the sample matrix has been filtered. Record each volume filtered in the 'Addns' column.

f. After all of the sample's volumes have been filtered, rinse the graduated cylinder. Rinse sufficiently to ensure all of the solids inside the graduated cylinder are transferred to the filtration apparatus.

(1) If the entire volume was used for the analysis, then rinse the inside of the sample container with at least three 10-mL portions of reagent water and transfer the rinses to the filtration apparatus. Rinse sufficiently to ensure all of the solids inside the bottle are transferred to the filtration apparatus.

g. Wash the filter with at least three 10 mL portions of reagent water when the sample matrix allows. Allow complete drainage between washings. If complete filtration takes more than 10 minutes, reanalyze the sample decreasing the sample volume.

h. If necessary, perform a final rinse to remove any residue from the sides of the filtration unit.

i. Continue suction at least 3 minutes after filtration is complete.

NOTE: This step is important to ensure that dissolved solids are rinsed through, and not dried in the filter.

9. The total sample volume filtered ('Vol. Used' column on the raw data worksheet) is calculated by adding all the volumes from the 'Initial Vol'. and 'Addns.' columns. Record the total sample volume used in the 'Vol. Used' column on the datasheet.

10. Transfer the filter back into its aluminum carrying dish and place in an oven maintained at 104° ± 1°C.

11. Record the oven temperature on the worksheet.

12. Dry the filter for the appropriate amount of time according to the analysis selected.

a. Analysis #10457 - dry the filter for at least one hour.

b. Analysis #13858 - dry the filter for at least 16 hours.

13. After the drying time, transfer the aluminum dish into a desiccator and cool to balance temperature.

14. After cooling, weigh the filter and record the oven dried weight of the filter.

    a. Analysis #10457 - repeat this drying desiccating and weighing cycle until weight change is less than 4% of the previous weight or ≤ 0.0005g, whichever is less.

    b. Analysis #13858 - require only one weight after a 16-hour drying cycle.

B. Analysis #14115

1. If pH is requested, analyze the sample for pH first and return the volume used for pH to the original sample bottle before starting the total volume test.

2. Measure the total volume of sample in a graduated cylinder.

The minimum acceptable volume is 100-mL.

3. Measure two equal aliquots using the entire volume of sample (i.e. for 1000 ml sample use two 500 ml aliquots; for 250 ml sample use two 125 ml aliquots).

4. Use a separate filtration apparatus for each aliquot.

5. Filter both aliquots for 6 hours or until no more liquid is readily obtained.

    a. If at least 500–mL have been filtered before 6 hours, filtration can be stopped.

    b. If necessary sample can be divided into four 250-ml aliquots in four funnels.

6. The entire sample is to be filtered.

7. Measure and record the total volume of all filtrate collected from the sample.

Calculations

A. Analysis #10457 and Analysis #13858:

$$mg\ Total\ Suspended\ Solids / L = \frac{(A - B) \times 1000 \times 1000}{Sample\ Volume\ (mL)}$$

Where:

A = Weight of filter, (in grams) after drying

B = Tare weight of filter (in grams)

B. Analysis #14115:

$$Ratio = \frac{Filtrate\ Volume\ (mL)}{Total\ Volume\ (mL)}$$

Multiply the ratio by 100 to obtain the percentage.

Statistical Information/Method Performance

Generate the method detection limit (MDL) and limit of quantitation (LOQ) annually by following the procedure in *QA-SOP11892* . The department maintains annual study data and updates to LIMS are made as needed by the QA department.

Generate the quality control acceptance windows by following the procedure outlined in *QA-SOP11896*. The limits are evaluated annually and updated as needed. These limits are maintained in LIMS for the relevant analysis number(s).

Quality Assurance/Quality Control

Batch size is limited to 20 samples or less.

No quality assurance procedures are required for Analysis #14115.

One method blank and one 150–mg/L LCS must be analyzed with each TSS batch.
A sample matrix duplicate must be analyzed with each group of not more than 10 samples.

Duplicate determinations should agree within 5% of their average weight.

When sample volume does not permit the analysis of the required duplicate(s), then a 150 mg/L LCSD must be analyzed to demonstrate method precision.

The maximum residue limit is 0.2 gram.

Samples from West Virginia require a usable precision measurement for each batch.

Analyzing one of the following on a batch containing a West Virginia sample fulfills this requirement: a non-program deleted duplicate or an LCSD.

See LIMS for current quality control (QC) acceptance windows. Follow guidelines in *T-WC-WI10360* for outlier QC data.

Attachment I

TSS Pre-Screen Sieving

1. Mark level of the liquid on the side of the sample container.

2. In a fume hood, place the ½" sieve on the ¼" sieve. Place both sieves on the bucket.

3. Pour entire sample through the sieves into the bucket.

4. Remove sieves and measure the weight of the residue collected on each sieve by scraping the residue into a specimen cup on a tared pan balance.

5. Record weights to 0.1g.

6. Mix sieved sample in the bucket and transfer 500 ml to a new plastic bottle.

7. Label and retain for TSS analysis.

8. Discard remaining screened (sieved) sample.

9. Fill original sample container with tap water to line previously marked.

10. Measure the volume of the tap water and record the total sample volume received.

11. Record the weight of the residue (½" sieve), weight of the residue (¼" sieve) and total volume sieved in the TSS analysis comments, including the following sentence: "This sample was sieved prior to the TSS analysis." Use analysis comment #9388.

QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits
T-WC-WI10359 Instructions for Collecting Data on the LLENS System
T-WC-WI10610 Fixed Suspended Solids (Gravimetric) (#207) Volatile Suspended Solids (Gravimetric) (#208) by SM 2540 E - 2011 or EPA 160.4 in Water

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End of document

Version history

Version	Approval	Revision information
10	28.SEP.2016	
11	10.DEC.2018	

E-11

Separatory Funnel Extraction by Method 3510C for BNAs by 8270 SIM in Wastewater (Rev. 9; 03/20/2019; T-OE-SVOA-WI10931)

eurofins	Always check on-line for validity. **Separatory Funnel Extraction by Method 3510C for BNAs by 8270 SIM in Wastewater**	Level: **Work Instruction**
Document number: **T-OE-SVOA-WI10931**		
Old Reference: **1-P-QM-WI -9015121**		
Version: **9**		Organisation level: **5-Sub-BU**
Approved by: **UKL3** Effective Date **20-MAR-2019**	Document users: **5_EUUSLA_GC/MS Semivolatiles_Manager, 5_EUUSLA_Organic Extraction_Manager, 6_EUUSLA_ Organic Extraction_Semivolatile Waters, 6_EUUSLA_GC/MS Semivolatiles_Analysts**	Responsible: **5_EUUSLA_Organic Extraction_Manager**

LIMS ID

Analysis 11012, 10465, 10466, 10470, 10471, 11912

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Revision Log

Revision **09**		**Effective Date:**	**This version**
Section	**Justification**		**Changes**
Revision Log	Formatting requirement		Removed revision logs up to the previous version
Throughout document	Update		Replaced EtQ references with D4 references and added hyperlinks
Procedure A.1,2	Reflects current practice		Updated to reflect use of one full bottle for samples and QC.
Procedure A.3	Reflects current practice		Added that the sample bottle needs to be etched.

Revision 09	**Effective Date:**	**This version**
Procedure E,F	Reflects current practice	Re-ordered steps so pH adjusting is before the addition of solvent.
Procedure E.1,2 and O.1,2	Reflects current practice	Added reference to *T-OE-GEN-FRM23822* for information on adjusting and checking PH.
Procedure J	Reflects current practice	Changed RPM from 2500 to 3000
Procedure L	Reflects current practice	Clarified that rinsing of beaker is for full liter samples only
Procedure P	Reflects current practice	Updated rinse volume from 15mL to 5mL
Procedure R1	Reflects current practice	Added wording to include the rinsing of the sides of the Rapid-Vap tube.
Procedure R2	Reflects current practice	Added wording to clarify how extract is brought to a final volume of 1 mL

Revision: 8	Effective Date:	30-SEP-2016
Section	Justification	Changes
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflects current analysis numbers	Added analysis numbers 14243, 14244, 14245
Apparatus and Equipment	Reflects current practice	Added Automated Water Extraction Bench, Rapid-Vap Evaporator, Rapid-Vap Concentrator Tubes
Procedure G.1. and G.2.	Reflects current practice	Added spike and surrogate details for new analysis numbers
Procedure L.-X.	Reflects current practice	Separated 1L extraction from 250-mL extraction and added new procedure for 250-mL extraction.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3510C, Rev 3, December 1996.
2. *Chemical Hygiene Plan,* current version.

Cross Reference

Document	Document Title
Analysis #*8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971* (***T-SVOA-WI9995***)	Semivolatiles by Methods 8270C/D SIM
Analysis #*10242, 10262, 11305, 11597* (***T-SVOA-WI9252***)	Determination of Parent and Alkyl Substituted Polynuclear Aromatic Hydrocarbons (PAHs), Alkanes and Geochemical Biomarkers by Gas Chromatography/Mass Spectrometry (GC/MS-SIM)
T-SVOA-WI11998	Semivolatile Spiking and Calibration Standards

Document	Document Title
T-OE-GEN-WI10864	Glassware Cleaning for Organic Extractions
T-OE-GEN-WI10876	Organic Extraction Standards Storage and Handling
T-OE-GEN-FRM23822	Instructions for checking PH during Extraction Procedures

Scope

This method is for the extraction of semivolatile organic compounds at low ppb to low ppm levels in water and wastewater matrices that are not prone to emulsions. High levels of organic compounds and/or extreme alkalinity or acidity in the sample often interfere with normal detection limits.

Basic Principles

A portion of sample is placed into a separatory funnel. Surrogate standards are added to each sample to monitor recovery. The pH of the sample is adjusted to >11 and the sample is serially extracted with methylene chloride. The pH of the sample is then adjusted to <2 and the sample is extracted with methylene chloride. The solvent fractions are combined, and the extract is dried and concentrated.

Procedural changes to use a reduced sample aliquot while maintaining the default reporting limits are permitted as long as the reagent aliquots are reduced proportionally and a quad study is performed and on file.

Reference Modifications

Surrogate and matrix spiking solutions are not added before the transfer to the separatory funnel for several reasons:

1. Samples must be poured from the amber bottles to determine the matrix and volume of sample to use for each extraction.

2. Many sample bottles have no headspace and there is no room to add surrogate to the sample in the bottle.

3. Due to the volume of samples extracted, a separate graduated cylinder for each sample is unrealistic.

4. To maintain consistency with all extractions, no samples are spiked in the bottle or graduated cylinders.

Interferences

Impurities in solvents, reagents, glassware, or other hardware used in sample processing lead to interferences with the method. All glassware must be rinsed with solvent before use. A method blank

is performed with each batch of sample to demonstrate that the extraction system is free of contaminants.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Each chemical compound must be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Extracts are concentrated on a steam bath; caution must be exercised while working around this apparatus.

All solvent waste generated from this preparation must be collected for recycling (if applicable) or disposed of in the designated containers. These are transferred to the lab-wide disposal facility. Any solid waste material (disposable pipettes, broken glassware, pH paper) is disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the technicians training records.

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the extraction and analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples, or one blind sample.

Sample Collection, Preservation and Handling

Samples are collected in glass bottles with PTFE-lined lids and stored at 0° to 6°C, not frozen, prior to extraction. Samples must be extracted within 7 days of sample collection. The extract is stored in an amber autosampler vial in the freezer =-10 °C for up to 40 days prior to analysis.

Apparatus and Equipment

1. 2-L separatory funnel for extracting organic components from an aqueous matrix

2. Kuderna-Danish (K-D) assembly with appropriate ampule for extracting the solvent used during the extraction

3. Water bath – VWR/LLI Model #1127 or equivalent

4. Graduated cylinders – Class A, assorted sizes

5. Pipettes – Class A, assorted sizes

6. Pipettes – Disposable

7. Solvent pumps – Brinkman, adjustable

8. Balance – Capable of weighing to 0. 1 g

9. Automatic shaker – Capable of holding 2 L separatory funnels

10. Centrifuge – Beckman GS-6 or equivalent

11. Sodium sulfate columns

12. Micro-snyder columns

13. Wash bottles – Teflon

14. Amber autosampler vials

15. Teflon boiling chips

16. Teflon separatory funnel

17. pH paper – Wide range

18. Automated Water Extraction Bench – capable of holding separatory funnels for 250-mL extractions

19. Rapid-Vap Evaporators – for concentration of 250-mL extractions

20. Rapid-Vap Evaporator Tubes – Class A

Reagents and Standards

1. Methylene chloride (CH_2Cl_2) – Pesticide grade or equivalent. Store at room temperature for up to one year.

2. 10N Sodium hydroxide (NaOH) – Lab chem or equivalent. Store at room temperature for up to one year.

3. Sulfuric acid – ACS grade or equivalent, concentrated. Store at room temperature for up to 1 year.

4. Sodium sulfate (Na2SO4) – Granular anhydrous reagent grade or equivalent. Bake at approximately 400°C for a minimum of 4 hours in a shallow pan prior to use to remove organic contaminants. After baking, store in a glass jar at room temperature for up to 1 year.

5. All QC standards added during extraction process are prepared by Organic Extractions using instructions generated by the standards database. Detailed instructions can be found in the corresponding analytical Analysis #*8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971*, 14243, 14244, 14245 and Analysis #*10242, 10262, 11305, 11597*.

Preparation of Glassware

See ***T-OE-GEN-WI10864***

Calibration

Not applicable to this procedure.

Procedure

A. Determine the volume of sample to be used for each extraction. Ideally, one full bottle is used. (Typically this is 1L for routine extraction or 250-mL for mini-extractions).

1. 1 Liter analysis numbers: *8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971*, *10242, 10262, 11305, 11597*.

2. 250 mL analysis numbers: 14243, 14244, 14245

3. Etch the outside of the sample bottle with a scriber at the meniscus.

4 . Shake the bottle vigorously.

5. Pour the sample into a separatory funnel.

6 . Record any comments about the sample on the extraction sheet.

NOTE: Reduced volume of sample is only used with prior approval of the client or if the matrix of the sample does not allow the extraction to proceed using full volume. The sample must then be diluted to 1 L (or 250 mL) with DI water.

B. The method blank, laboratory control sample (LCS), and laboratory control sample duplicate (LCSD) (if applicable) are prepared using 1 L (or 250 mL for mini-extractions) of non-deionized reagent water (tap water) measured into the separatory funnel.

C. The background, MS, and MSD (if applicable) are prepared on three separate aliquots of a field sample.

D. Surrogates and Spiking Solutions

1. Surrogates

a. Analysis 0039, 8357, 10010, 10262, 10137, 11305, and 11913 - Add 1.0 mL BNA surrogate to all samples, blanks, LCS, LCSD (if applicable), MS, and MSD (if applicable).

b. Analysis 14243, 14244, and 14245 – Add 1.0 mL Mini BNA Surrogate to all samples, blanks, LCS, LCSD (if applicable), MS, and MSD (if applicable).

NOTE: Be certain the standard drips directly into the sample without touching the glass side of the separatory funnel to avoid poor recoveries.

2. Spiking solutions

a. Analysis 0039, 8357, 10137 and 11913 - Add 1.0 mL of SIM spiking solution to the LCS, LCSD (if applicable), MS, and MSD (if applicable).

b. Analysis 10010 – Add 1.0 mL of SIM spiking solution to the LCS, LCSD (if applicable) MS, and MSD (if applicable). Spike a separate LCS, LCSD with 1.0 mL of Benzenethiol spiking solution.

c Analysis 10262 – Add 1.0 mL of Alkyl PAH spike to the LCS, LCSD (if applicable) MS, and MSD (if applicable).

d. Analysis 11305 – Add 1.0 mL of DPnB spiking solution to LCS, LCSD if applicable, MS and MSD.

e. Analysis 14243, 14244, 14245 – Add 1.0 mL of Mini SIM spiking solution.

f. If the sample requires additional semivolatile compounds, 1.0 mL of 1 ppm spike (or 0.25 mL for 250-mL extractions) of this compound is added at this time.

g. See analysis SOPs for spike details.

3. See ***T-SVOA-WI11998*** for preparation of spikes and standards.

4. See ***T-OE-GEN-WI10876*** for storage and handling of spikes.

E. Adjusting pH of samples to >11

NOTE: If samples are entered for 8357, 10262, 11305, or 11913 proceed to Procedure O.

1. For 1 L extractions: See document ***T-OE-GEN-FRM23822*** for instructions on checking the PH of samples.

a. Add approximately 12 drops of 10N sodium hydroxide to Method Blank, LCS/LCSD, and samples using a disposable Pasteur pipette. Swirl samples to mix.

b. Check pH of samples using Hydrion 10 to 12 range pH paper or equivalent to check if pH of >11 has been attained. All pH checks are performed by using a glass disposable pipet to withdraw a few drops of sample and transferring the sample to the pH paper.

NOTE: If a pH of >11 is not observed additional 10N sodium hydroxide should be added by adding 6 to 7 drops using a disposable Pasteur pipette, swirling, and re-checking the pH with Hydrion 10 to 12 range pH paper or equivalent. Please see a supervisor if a pH of >11 is **not** achieved after repeating the 10N sodium hydroxide addition 5x. Please note any issues on the batch log for any samples that are difficult to pH adjust.

2. For 250 mL extractions: See document ***T-OE-GEN-FRM23822*** for instructions on checking the PH of samples.

a. Add approximately 4 drops of 10N sodium hydroxide to samples, Method Blank, and LCS/LCSD using a disposable Pasteur pipette. Swirl samples to mix.

b. Check pH of samples using Hydrion 10 to 12 range pH paper or equivalent to check if pH of >11 has been attained.

NOTE: If a pH of >11 is not observed additional 10N sodium hydroxide should be added by adding 2 to 3 drops using a disposable Pasteur pipette, swirling, and re-checking the pH with Hydrion 10 to 12 range pH paper or equivalent. Please see a supervisor if a pH of >11 is **not** achieved after repeating the 10N sodium hydroxide addition 5x. Please note any issues on the batch log for samples that are difficult to pH adjust.

F. Determine the volume of sample used for extraction:

1. If the original sample bottle is empty:

   a. Use a solvent pump to measure 60 mL of methylene chloride (15 mL for 250-mL extractions) and rinse the sample bottle by capping and inverting several times prior to adding the methylene chloride to the separatory funnel.

   b. Transfer the solvent to the separatory funnel.

   c. Fill the bottle to the etched line with non-deionized reagent water (tap water) and pour into a graduated cylinder to determine the initial volume.

2. Alternatively, tare the empty sample bottle on the balance.

   a. Fill the bottle with non-deionized reagent water (tap water) to the etched line and place the bottle back onto the balance.

   b. Round the weight to a whole number. This is the volume of sample used for extraction.

3. If the sample container is not empty, use a solvent pump to measure 60 mL of methylene chloride (15 mL for 250-mL extractions) and add the solvent directly to the separatory funnel.

G. Cap the funnel, invert, and vent immediately.

H. Handshake and vent frequently until the pressure is stable.

I. For full liter analyses *8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971*, *10242, 10262, 11305, 11597*: Place the sample on the automatic shaker and shake at the designated speed for 2 minutes with the stopcocks closed.

For mini analyses 14243, 14244, 14245: Separatory funnels remain on the Automated Water Extraction Bench. Press the green "start" button to lower the hood and activate the Automated Water Extraction Bench. The bench will tumble for 2 minutes. The sash will rise after the samples have been tumbled.

NOTE: Shaker speeds vary greatly between instruments; the proper setting is marked on each.

J. For full liter analyses *8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971*, *10242, 10262, 11305, 11597*: Place the separatory funnel on the rack and allow it to sit undisturbed for approximately 10 minutes. For mini analyses 14243, 14244, 14245, the separatory funnel will sit undisturbed on the Automated Water Extraction Bench.

The time required for extracts to set undisturbed is based upon visual confirmation that the layers are adequately separated. Additional time may be necessary for samples with unusual high density (i.e. high salt content).

If an emulsion forms and is >1/3 of the volume of the solvent layer, mechanical techniques such as pouring the emulsion into a Teflon separatory funnel and centrifugation (3000 rpm for 2 minutes) must be employed to complete the separation.

K. For full liter analyses *8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971*, *10242, 10262, 11305, 11597*: Drain the solvent layer into a metal beaker and pour it through approximately 10 cm (~4 inches) of sodium sulfate into a K-D apparatus containing Teflon™ boiling chips. For mini analysis 14243, 14244, 14245, drain the solvent layer directly into the sodium sulfate column containing approximately 5 cm (~2 inches) of sodium sulfate. This drains into a Rapid-Vap Evaporator tube.

L. Full liter analyses only: Rinse the metal beaker with approximately 20 mL of methylene chloride and pour through salt column.

M. Use a solvent pump to add 60 mL of methylene chloride (15 mL for 250-mL extractions) to the separatory funnel and repeat Procedure steps I through K, venting only as necessary.

N. Again, use a solvent pump to add 60 mL of methylene chloride (15 mL for 250-mL extractions) to the separatory funnel and repeat Procedure steps I through K, venting only as necessary. The acid extract is also added to this KD.

O. Adjusting pH of sample to <2

1. For 1 L extractions: See document ***T-OE-GEN-FRM23822*** for instructions on checking the PH of samples.

   a. Add approximately 1mL (1/2 full disposable Pasteur pipette) of concentrated sulfuric acid to samples and swirl to mix.

   b. Check pH of samples using Hydrion 1 to 2.5 range pH paper or equivalent to check if pH of <2 has been attained.

NOTE: If a pH of <2 is not observed additional concentrated sulfuric acid should be added by adding 10 to 11 drops using a disposable Pasteur pipette, swirling, and re-checking the pH with Hydrion 1 to 2.5 range pH paper or equivalent. Please see a supervisor if a pH of <2 is **not** achieved after repeating the concentrated sulfuric acid addition six times. Please note any issues on the batch log for samples that are difficult to pH adjust.

2. For 250 mL extractions: See document ***T-OE-GEN-FRM23822*** for instructions on checking the PH of samples.

a. Add approximately 12 drops of concentrated sulfuric acid to samples, Method Blank, LCS/LCSD using a disposable glass pipette. Swirl samples to mix.

b. Check pH of samples using Hydrion 1 to 2.5 range pH paper or equivalent to check if pH of <2 has been attained.

NOTE: If a pH of <2 is not observed additional concentrated sulfuric acid should be added by adding 3 to 4 drops using a disposable Pasteur pipette, swirling, and re-checking the pH with Hydrion 1 to 2.5 range pH paper or equivalent. Please see a supervisor if a pH of <2 is **not** achieved after repeating the concentrated sulfuric addition six times. Please note any issues on the batch log for samples that are difficult to pH adjust.

For full liter analyses *8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971*, *10242, 10262, 11305, 11597*: Rinse metal beaker with approximately 20 mL of methylene chloride. Pour the solvent from the metal beaker into the sodium sulfate column. For mini analyses 14243, 14244, 14245, drain the solvent layer directly into the sodium sulfate column.

P. Use a wash bottle to rinse the sodium sulfate column with approximately 20 mL of methylene chloride (5 mL for 250-mL extractions).

Q. For 1-Liter extractions

1. Attach a 3-ball Snyder column to the K-D and concentrate the extract to approximately 1-mL on a steam bath at 80º to 90ºC. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. Adjust the vertical position of the apparatus and the water temperature as needed to complete the concentration in 10 to 20 min.

2. Allow the sample to cool for 10 minutes.

3. Microsnyder the sample to slightly below 1 mL.

4. Bring the combined extract to 1.0 mL using methylene chloride.

   a. Determine the final volume by placing the extract into an amber autosampler vial and comparing the level in the vial to a reference vial containing the exact targeted final volume. Add methylene chloride to the extract with a disposable pipette until exactly the same level is in both vials.

If too much solvent is added to the sample vial, remove the extract from the vial and concentrate it by microsnydering to slightly less than the targeted final volume and rebottle.

b. Cap securely and store in the freezer.

c. Record the final volume on the extraction log.

R. For 250-mL Extractions

1. Place the evaporator tube containing the solvent extract into the pre-heated (approximately 90° C) Rapid-Vap Evaporator. Set the timer for 10 minutes and speed at 75%(the speed is the rotation within the unit while the sample is evaporating). Confirm that the nitrogen is set at 15 psi (this is adjusted on the regulator attached to the nitrogen line). Allow the samples to concentrate to around 0.5 mL. Periodically rinse the sides of the RapidVap tube with methylene chloride during the concentration.

NOTE: The temperature, timer, and speed settings can be adjusted as necessary to accommodate difficult sample matrices, etc.

2. Mix the sample thoroughly in the Concentrator Tube and transfer the extract to an amber autosampler vial. The extract is quantitatively transferred from the evaporator tube using a Pasteur pipette to a 1mL amber autosampler vial using methylene chloride. The final volume of 1mL is quantified by comparing the extract auto sampler vial to a reference auto sampler vial containing exactly 1mL of methylene chloride. Cap the extract securely and store in the freezer. Record the final volume on the extraction log.

Calculations

See analysis method.

Statistical Information/Method Performance

See analysis method.

Quality Assurance/Quality Control

A batch is defined as the samples to be extracted on any given day but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. For each batch of samples extracted, a blank, an LCS, an MS, and MSD must be extracted. If insufficient volume of sample is available for MS/MSD, then an LCSD must be prepared instead. Also, if the batch contains only field or equipment blank samples, the LCS/LCSD QC pairing must be used.

If any client, state, or agency has more stringent QC or batch requirements, these must be followed instead.

T-OE-GEN-FRM23822 Instructions for Checking pH During Extraction Procedures
T-OE-GEN-WI10864 Glassware Cleaning for Organic Extractions
T-OE-GEN-WI10876 Organic Extraction Standards Storage and Handling
T-SVOA-WI11998 Semivolatile Spiking and Calibration Standards
T-SVOA-WI9252 Determination of Parent and Alkyl Substituted Polynuclear Aromatic Hydrocarbons (PAHs), Alkanes and Geochemical Biomarkers by Gas Chromatography/Mass Spectrometry (GC/MS-SIM)
T-SVOA-WI9995 Semivolatiles in Waters and Soils by Methods 8270C/D SIM by GC/MS

End of document

Version history

Version	Approval	Revision information
8	30.SEP.2016	
9	20.MAR.2019	

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E-12

Semivolatiles in Waters and Soils by Methods 8270C/D SIM by GC/MS
(Rev. 14; 03/21/2019; T-SVOA-WI9995)

LIMS ID

Analysis DOD - 8357, 0038, 0039, 10010, 10137, 10138, 10725, 11915, 11917, 12969, 12970, 12971, 11913, 14243, 14244, 14367, 14374, 14962

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Revision Log
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Basic Principles
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Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
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Table I
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Revision Log

	Revision: 14	**Effective Date: This version**
Section	**Justification**	**Changes**
Title	Enhancement	Updated title to include matrix and technology
LIMS ID	Enhancement	Updated to add analyses that were recently activated
Reference	Reflects current practice	Removed reference to out of date 8000 Method
Cross Reference	Typographical error	Updated document to *T-SVOA-WI9594*
Scope	Enhancement	Added information regarding where to find analytes offered
Sample Collection, Preservation, and Handling	Enhancement	Added information regarding sample collection containers
Reagents and Standards	Reflects current practices	Many updates made throughout section to reflect current practices
Calibration	Enhancement	Removed redundant verbiage
Procedure A. Standard Preparation	Clarity and reflects current practices	Many updates made throughout section for clarity and to reflect current practice.
Quality Assurance/Quality Control	Enhancement	Added how many samples can be included on each batch. Added acceptance limits for MS/D
Quality Assurance/Quality Control	Enhancement	Added troubleshooting options for surrogate failures
Table IV	Reflects current practice	Changed 1,2-Diphenylhydrazine to N-Nitrosodiphenylamine
Figure 1	Redundant information	Removed Figure to be consistent with other SOPs
Reference	Most current version	Updated SW-846 8270D revision date
Sample Collection, Preservation, and Handling	Enhancement	Changed working to match other 8270 methods
Scope	Enhancement	Adjusted wording to include more than just PAHs
Calibration C	Enhancement	Adjusted wording to clarify that the minimum number of standards for a linear curve fit is five
Calibration D	Reflects current practices	Clarified the CCV standard concentration

	Revision: 14	**Effective Date:** **This version**
Section	**Justification**	**Changes**
Procedure B	Reflects current practices	Added 8270C tuning criteria
Procedure C	Enhancement	Clarified that parameters can be adjusted if needed
Procedure D	Enhancement	Clarified the software used for data analysis
Procedure E	Reflects current practices	Updated target identification working for qualitative analysis
Quality Assurance/ Quality Control	Enhancement	Changed references to "PAHs" to "target compounds"

Revision: 13	**Effective Date:**	Aug 23, 2017
Section	**Justification**	**Changes**
Throughout document	Reflect new document numbers	Updated document references to D4 numbers
References	Most current version	Added reference for Method 8000D
Calibration C	Enhancement	Added % error verification for initial calibration levels
Procedure B	Reflects current practices	Updated tuning procedure requirements
Calculations	Enhancement	Added calculation for % Error
Quality Assurance/Quality Control	Reflects current practices	Added LLOQ information

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 8270D (SIM), July 2014.
2. Test Methods for Evaluating Solid Wastes, SW-846 Method 8270C (SIM), December 1996.
3. Test Methods for Evaluating Solid Wastes, SW-846 Method 8000D, July 2014.
4. *Hewlett-Packard Operations Manuals.*
5. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Q-EQA-FRM6869	Nonconformance Form

Document	Document Title
T-SVOA-WI9598	GC/MS Preventative and Corrective Maintenance
T-SVOA-WI9594	GC/MS Audit Process
QA-SOP11178	Demonstrations of Capability

Scope

This method is suitable for the determination of low–level semivolatile compounds from soils and waters by selected ion monitoring (SIM) Gas Chromatography/Mass Spectrometry (GC/MS). The analysis applies to a concentration range that spans from an MDL of 0.01 µg/L in water (0.33 µg/kg for soil) to an upper calibration concentration of 10 µg/L in water (330 µg/kg for soil). Specific compound lists and their associated MDLs/LOQs are found in the Laboratory Information Management System (LIMS) under the analysis numbers listed on the front page of this SOP.

Basic Principles

An environmental sample (soil, water, sludge, etc.) is solvent extracted and then analyzed by electron impact gas chromatography/mass spectrometry (EI GC/MS). By analyzing for specific masses over a narrow mass range, SIM analysis allows for longer dwell times at specific masses. This allows for significantly greater sensitivity of analysis with a small reduction in specificity of analysis.

Interferences

Sample matrices can have an effect on the ability of the GC/MS system to resolve the individual masses used for quantification. Samples may require dilutions or a reduction in the extraction volume/weight used in order to achieve proper target separation and determination.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.
The toxicity of each reagent has not been precisely determined.

However, each reagent should be treated as a potential health hazard. Safety measures would include the use of fume hoods, safety glasses, lab coats, and gloves.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of percent recovery. Various options are available for a DOC and can include four laboratory control samples, one blind sample, or one ICAL with ICVs and/or CCVs. Refer to *QA-SOP11178* for more guidance on these options.

Sample Collection, Preservation, and Handling

Water samples may be preserved with sodium thiosulfate (Na2S2O3) and must be extracted within 7 days of collection. Solid samples are not preserved and must be extracted within 14 days of collection.

Water samples are collected in 1L or 250mL glass amber containers. Soils are collected in 250mL glass containers.

Samples are stored at 0º to 6ºC, not frozen. Extracts must be analyzed within 40 days of extraction and are stored in amber vials at ≤ -10C (freezer).

Apparatus and Equipment

1. Hewlett-Packard Model 5890 Gas Chromatograph or equivalent

2. Hewlett-Packard Model 5971, 5972, or Agilent Model 5973 Mass Selective Detector or equivalent

3. Hewlett-Packard Chemstation Software, Thru-Put Systems Target data system or equivalent

4. Volumetric flask – Assorted sizes

5. Hamilton Gastight syringe – Assorted sizes

Reagents and Standards

1. Tune Check Solution – 500 ng/µL solution of decafluorotriphenylphosphine (DFTPP) containing pentachlorophenol, benzidine and p,p'–dichlorodiphenyltrichloroethane (DDT) in methylene chloride, Absolute Standards Inc. Catalog #43030, or equivalent; store at 0° to 6°C, not frozen

2. Methylene chloride – (MeCl2 or CH2Cl2) – Baker Ultra Resi–Analyzed, or equivalent

3. Internal standard solution – 2000 ppm in methylene chloride Restek custom Catalog #A073516, or equivalent

4. Absolute PAH Standard – 1000 ppm solution of PAHs in methylene chloride, Catalog #93462, or equivalent

5. 8270 Surrogate standard solution – 1000 ppm solution in methylene chloride, Restek custom Catalog #A0114172, or equivalent

6. PAH Plus Methylnaphthalenes - 2000 ppm in methylene chloride, Phenova Catalog #AL0-101233, or equivalent

7. Custom BNA Mix 1 - 1000 ppm in methylene chloride, Phenova Catalog #AL0-130057, or equivalent

8. Custom BNA Mix 2 - 1000 ppm in methylene chloride, Phenova Catalog #AL0-130058, or equivalent

9. Quinoline – 1000 ppm in methylene chloride, Absolute Standards Catalog #70353, or equivalent

10. Hexachlorobenzene – 1000 ppm in acetone, Absolute Standards Catalog, #70195, or equivalent

11. Bis(2-Chloroethyl)ether – 1000 ppm in methylene chloride, Absolute Standards Catalog #70075, or equivalent

12. EPA Method 8070 – Nitrosamines - 2000 ppm in Methanol, Absolute Standards Catalog #19222 or equivalent

13. EPA Method 606 – Phthalates – 2000 ppm in Methanol, Absolute Standards Catalog # 19242 or equivalent

14. 1,4-Dioxane – 2000 ppm in methanol, Absolute Standards Catalog #90871 or equivalent

NOTE: Special compounds may be added to spikes and calibration standards as necessary. These compounds and concentrations are determined by the analyst and added in appropriate proportions to achieve proper concentrations. If a modification to a solution's final volume is required, adjust the volume of stock solutions added to achieve the appropriate concentrations.

Calibration

A. Calibration is accomplished using an internal standard calibration technique. Calibration standards at five or more concentration levels are analyzed. The relative response factor is calculated for each compound at each concentration level. The relative standard deviation of the response factors determines the suitability of the average relative response factor for calculation of concentration.

1. If the performance criteria are met for the DFTPP solution (from Procedure step B.), perform an initial calibration by analyzing five or more calibration standard solutions referenced previously.

2. After the calibration standard analyses, inject the MDL/LOQ solution. The GC/MS system must be able to detect the compounds in the MDL/LOQ solution. If the system does not detect the compounds, then the tuning and calibration procedure must be repeated under conditions that will yield a detection for the compounds in the MDL/LOQ solution. Instrument maintenance as outlined in *T-SVOA-WI9598* may be required.

3. After the MDL/LOQ solution has been run, inject the ICV solution. This standard must be analyzed after the initial calibration standards and before any samples are analyzed.

B. Calculate the relative response factor for each compound and surrogate for each calibration solution. Refer to the Calculations Section for equation.

C. For each compound and surrogate, calculate the average RRF from the RRFs of the calibration standards. For 8270D, each compound must meet the minimum response factor criteria listed in Table III. Refer to the Calculations Section for equation.

For 8270C, if the %RSD of the response factors is less than or equal to 15%, then the average relative response factor $(\overline{RRF})$ is used for quantitation. If the %RSD exceeds 15% then an alternate curve fit must be used for quantitation. A linear curve fit is a suitable alternative when 5 or more calibration standards have been analyzed.

For 8270D, if the %RSD of the response factors is less than or equal to 20%, then the average relative response factor is used for quantitation. If the %RSD exceeds 20% then an alternate curve fit must be used for quantitation. A linear curve fit is a suitable alternative when 5 or more calibration standards have been analyzed.

The % Error is measured between the calculated and expected amounts of an analyte at each calibration level to determine the calibration function acceptability for linear and non-linear curves. Refer to the Calculations Section for equation. The % Error should be ≤ 30% for all standards. For some data uses, ≤ 50% may be acceptable for the lowest calibration level.

D. Continuing calibrations

Verify the MS tune and initial calibration at the beginning of every 12–hour work shift during which analyses are performed by analyzing a standard near the midpoint of the calibration.

1. The MS tune is verified following the instructions given in Procedure B. If the mass spectrum of the DFTPP peak does not meet the abundance criteria described in Table I for 8270C and Table II for 8270D, the mass spectrometer must be retuned until all criteria are met.

2. A continuing calibration verification (CCV) is analyzed after the tune each day a curve is not analyzed. For 8270D, each compound must meet the minimum response factor criteria listed in Table III. Refer to the Calculations Section for equation.

The % Drift for each compound should not be greater than ±20%. If the %Drift exceeds 20%, then corrective action must be taken and a new initial calibration may need to be performed.

3. The absolute areas of the quantitation ions of the internal standard must fall within –50% to +100% of the areas from the mid-level calibration standard produced during the last initial calibration. If the area for each internal standard is not within this window then corrective action must be taken and documented.

Procedure

The GC/MS is configured in the selected ion monitoring (SIM) mode with a total cycle time (including voltage reset time) of 1 second or less. The GC/MS system must be capable of meeting the following specifications:

Mass range:	35 to 500 amu
Scan time:	1 scan/sec
GC column:	30 m x 0.25 mm x 0.5 µm film Restek Rxi – 5 or equivalent
Injector temperature:	250° to 300°C
Transfer line temperature:	250° to 300°C
GC Oven:	Time and Temperature Programmable
EI condition:	70 eV
Mass scan:	Capable of SIM scanning (see manufacturer's instructions)
Carrier gas:	Helium at approximately 30 cm/sec.

A. Standard preparation

1. Internal standard

Internal standard mix is added to all standards and subsequent samples.

a. Soils and 1000 mL water extractions are at a concentration of 1 µg/mL. Using a 25-µl syringe, 10 µL of Restek Semivolatile Internal Standard Mix or equivalent prepared at a concentration of 100 µg/mL in methylene chloride is added to 1 mL of standard or sample extract.

Mix	Concentration (ppm)	Amount Added (µL) to 10 mL
Internal Standard	2000	500

b. 250 mL water extractions are at a concentration of 0.25 µg/mL. Using a 25-µl syringe, 10 µl of Restek Semivolatile Internal Standard Mix or equivalent prepared at a concentration of 25 µg/ml in methylene chloride is added to 1 mL of standard or sample extract.

Mix	Concentration (ppm)	Amount Added (µL) to 10 mL
Internal Standard	2000	125

2. Stock standard preparation

To prepare the 10–ppm working stock, using a Hamilton Gastight syringe, measure the following stocks into a 10–mL volumetric flask and dilute to volume with methylene chloride. See Figure 1 for a list of compounds. Stocks are prepared every 6 months or sooner, if comparison with the QC check samples indicates a problem.

Mix	Concentration (ppm)	Amount Added (µL) to 10 mL
PAH Standard	1000	100
Custom SIM Surrogate	1000	100
Quinoline	1000	100
Hexachlorobenzene	1000	100
Bis(2-Chloroethyl)ether	1000	100
Nitrosamines	2000	50
Phthalates	2000	50
1,4-Dioxane	1000	100

3. Calibration standards

Standard ID	Preparation	Final Volume
Level 1: LPAH0.01ppm	2 µL of 10–ppm working stock + 20 µL of 100–ppm internal standard	To 2.0 mL MeCl2
Level 2: LPAH0.05 ppm	10 µL of 10–ppm working stock + 20 µL of 100–ppm internal standard	To 2.0 mL MeCl2
Level 3: LPAH0.2 ppm	40 µL of 10–ppm working stock + 20 µL of 100–ppm internal standard	To 2.0 mL MeCl2
Level 4: LPAH0.5 ppm	250 µL of 10–ppm working stock + 50 µL of 100–ppm internal standard NOTE: this level to be used as CCV	To 5.0 mL MeCl2
Level 5: LPAH001 ppm		

Standard ID	Preparation	Final Volume
	500 µL of 100–ppm working stock + 50 µL of 100–ppm internal standard	To 5.0 mL MeCl2
Level 6: LPAH005 ppm	500 µL of 100–ppm working stock + 10 µL of 100–ppm internal standard	To 1.0 mL MeCl2
Level 7: LPAH010 ppm	1000 µL of 100–ppm working stock + 10 µL of 100–ppm internal standard	To 1.0 mL MeCl2
MDL/LOQ standard: 0.02 ppm	2 µL of 10–ppm working stock + 10 µL of 100–ppm internal standard	To 1.0 mL MeCl2
MDL/LOQ standard: 0.005 ppm	100 µL of 0.05–ppm calibration standard + 9 µL of 100–ppm internal standard	To 1.0 mL MeCl2

a. For samples extracted with miniature separatory funnels (250 mL), the calibration standards listed in the above table will be diluted 4x. The calibration range will be from 0.0125 µg/mL to 2.5 µg/mL. The MDL standard and ICV will also be diluted 4x.

b. One of the calibration standards must be at a concentration at or below the limit of quantitation (LOQ). The other concentrations correspond to the expected range of concentrations found in samples or should define the working range of the GC/MS system.

c. The concentration of the MDL standard may require modification dependent upon the current MDL values. Document the procedure concentrations and volumes in the SEMIS folder located on the shared drive of the network.

d. In addition to the calibration standards, initial calibration verification (ICV) standard must be prepared using a separate source from the working stock. The concentration of the ICV standard is at or around the mid–Level of the calibration curve. A PAH spike mix different from the working stock is prepared as indicated below. As referenced in *T-SVOA-WI9594* there must be an ICV standard form that compares the average relative response factors from the ICAL with the response factors from the ICV standard. The ICV recovery must be within ±30% or approval is required for those targets outside of this window. The ICV must be listed on all data package forms for the tune. The preparation of these standards is recorded in the SEMIS folder located on the shared drive of the network. The calibration standards are prepared as needed based on the frequency of analysis and contingent upon successfully passing continuing calibration check standards.

Mix	Concentration (ppm)	Amount Added (µL) to 10 mL
Internal Standard	100	100
PAH Plus Methylnaphthalenes	2000	5
Custom BNA Mix 1	1000	10
Custom BNA Mix 2	1000	10
N-Nitrosodiphenylamine	1000	10

e. All standards are stored at 0° to 6°C and protected from light.

B. Tuning

1. The DFTPP tune check standard is prepared at a concentration of 50 µg/mL. For samples extracted with miniature separatory funnels (250mL), the tuning solution is prepared at a concentration 4x lower.

Mix	Concentration (ppm)	Amount Added (µL) to 25 mL
CLP Tuning Mix	500	2500

Mix	Concentration (ppm)	Amount Added (µL) to 10 mL
CLP Tuning Mix	500	250

2. The DFTPP tune check standard is used to assess GC column performance and injection port inertness as well as mass spectrometer performance. The GC/MS system's tune is checked by inspecting the mass spectrum of the DFTPP peak and column performance and injection port inertness is evaluated with pentachlorophenol, benzidine, and DDT.

3. DFTPP must meet the criteria specified in Table I for 8270C and Table II for 8270D. If these criteria are met, standardization may begin at this point. If DFTPP fails the specified criteria, corrective action must be taken and the DFTPP reinjected until acceptable criteria are obtained. Tuning and DFTPP evaluation must take place every 12 hours that analyses are to be performed.

4. 8270D: DDT breakdown must be ≤ 20% and tailing factors for benzidine and pentachlorophenol must be ≤2. Note that DDT breakdown greater than 20 percent may be acceptable if you are calibrating for polynuclear aromatic hydrocarbon compounds only. Consult supervisor when this situation occurs.

8270C: DDT breakdown must be ≤ 20% and tailing factor for benzidine must be ≤ 3 and pentachlorophenol must be ≤ 5. Note that DDT breakdown greater than 20 percent may be acceptable if you are calibrating for polynuclear aromatic hydrocarbon compounds only. Consult supervisor when this situation occurs.

C. Method set-up

1. Prior to analysis by SIM (Selective Ion Monitoring) a check standard must be analyzed in the full scan mode to determine where the specific start and stop times are for each of the groups.

NOTE: Each program must be identical for the Temperature and Pressure programs so that the peak retention times are the same.

2. Each time column maintenance is performed, the scan times are reviewed to ensure that all ions are included. The instrument setup in the method is as follows. In the Set Acquisition Mode, select SIM. See specific Group for Dwell time in msec. Set the Low Res to "N" and the Start Time for each group. In the m/z field, enter the ions for each group from the following tables:

GROUP 1 (Dwell – 60 msec)			
Compound	***1.1.1.1 Type***	Quant Ion (m/z)	Monitor Ion (m/z)
1,4-Dioxane	Target	88	58
N-Nitrosodimethylamine	Target	74	42

GROUP 2 (Dwell – 40 msec)			
Compound	***1.1.1.2 Type***	Quant Ion (m/z)	Monitor Ion (m/z)
Quinoline	Target	129	102
Benzenethiol	Target	110	66
1,4-Dichlorobenzene-d4	ISTD	152	150
Naphthalene-d8	ISTD	136	68
Naphthalene	Target	128	129
Bis(2-chloroethyl)ether	Target	93	63

GROUP 3 (Dwell – 40-50 msec)			
Compound	***Type***	Quant Ion (m/z)	Monitor Ion (m/z)
Dimethylphthalate	Target	163	194
2-Methylnaphthalene	Target	142	141
1-Methylnaphthalene	Target	142	141
1-Methylnaphthalene-d10	Surrogate	152	
Acenaphthylene	Target	152	76
Acenaphthene-d10	ISTD	164	162
Acenaphthene	Target	154	152

GROUP 4 (Dwell – 25-30 msec)			
Compound	***Type***	Quant Ion (m/z)	Monitor Ion (m/z)
Dibenzofuran	Target	168	139
Diethylphthalate	Target	149	177
Fluorene	Target	166	165
Hexachlorobenzene	Target	284	142
N-Nitrosodiphenylamine	Target	169	168
Phenanthrene-d10	ISTD	188	94
Phenanthrene	Target	178	176
Anthracene	Target	178	176

GROUP 5 (Dwell – 50 msec)			
Compound	***Type***	Quant Ion (m/z)	Monitor Ion (m/z)
Di-n-butylphthalate	Target	149	150
Fluoranthene	Target	202	101
Pyrene	Target	202	101
Flouranthene-d10	Surrogate	212	106

GROUP 6 (Dwell – 40 msec)			
Compound	***Type***	Quant Ion (m/z)	Monitor Ion (m/z)
Butylbenzylphthalate	Target	149	91
Benzo(a)anthracene	Target	228	226
Chrysene-d12	ISTD	240	236
Chrysene	Target	228	226
Bis(2-ethylhexyl)phthalate	Target	149	167
Di-n-octylphthalate	Target	149	150
Benzo(b)fluoranthene	Target	252	253
Benzo(k)fluoranthene	Target	252	253
Benzo(e)pyrene	Target	252	253

GROUP 6 (Dwell – 40 msec)			
Compound	***Type***	Quant Ion (m/z)	Monitor Ion (m/z)
Benzo(a)pyrene	Target	252	253
Perylene-d12	ISTD	264	260
Benzo(a)pyrene-d12	Surrogate	265	132
Perylene	Target	252	253

GROUP 7 (Dwell – 100 msec)			
Compound	***Type***	Quant Ion (m/z)	Monitor Ion (m/z)
Indeno(1,2,3-cd)pyrene	Target	276	138
Dibenz(a,h)anthracene	Target	278	139
Benzo(g,h,i>perylene	Target	276	138

It is not required to use the exact parameters above as long as the performance required by the method is met.

D. Analysis of Samples

1. Analyze a predetermined aliquot of each sample extract under the same conditions used for the initial and/or continuing calibrations.

2. At the conclusion of data acquisition, use the data processing software to tentatively identify peaks within the retention time window of interest. Examine the ion abundances of components of the chromatogram. If the ion abundance of the main ion used for quantitation exceeds the calibration range, dilute the aliquot and reanalyze. When preparing dilutions, add sufficient internal standard to maintain the same concentration that was used.

E. Qualitative Analysis:

A compound is identified by comparison of the following parameters with those of a standard of this suspected compound (standard reference spectra). In order to verify identification, the following criteria must be met:

1. The intensities of the characteristic ions of the compound must maximize in the same scan or within one scan of each other.

2. The sample component relative retention time must compare within ± 0.06 RRT units of the RRT of the standard component.

3. The relative intensities of the qualifier ion(s) should agree within 30% of the relative intensities of these ions in the reference spectrum. The reference mass spectrum used for this comparison should be generated by the laboratory using the conditions of the method.

F. Quantitative Analysis:

When a compound has been identified, quantitation is based on the internal standard technique and the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. Refer to the Calculations Section for the equations.

Calculations

Calculation of the relative response factor (RRF):

$$RRF = \frac{[A(x) \times C(is)]}{[A(is) \times C(x)]}$$

Where:

A(x) = Area of the quantitation ion for the compound being measured
C(is) = Concentration of the specific internal standard
A(is) = Area of the quantitation ion for the specific internal standard
C(x) = Concentration of the compound being measured

Calculate the relative standard deviation:

$$\% RSD = \frac{SD}{\overline{RRF}} \times 100$$

Where:

$$SD = \sqrt{\frac{\sum (RF - avg\ RF)^2}{n-1}} \quad and\ \overline{RRF} = \frac{\sum RRF}{n}$$

Calculate the 1st Order (linear regression: Y = M(X) + B

Where:

Y = <u>Conc Std</u>

$$X = \frac{\text{Conc Inst}}{\text{Area Std}} \Big/ \text{Area Istd}$$

M = 1st degree slope
B = Y intercept

Calculation of the percent drift:

$$\%\ Drift = \frac{C(i) - C(c)}{C(i)} \times 100$$

Where:

C(i) = Calibration check compound standard concentration
C(c) = Measured concentration using selected quantification method

Quantitative Calculations

Waters:

$$Concentration\ (\mu g/L) = \frac{A(x) \times I(is)}{A(is) \times \overline{RRF}\ V(o)}$$

Where:

A(x) = Area of the quantitation ion for the compound to be measured
I(is) = Amount of internal standard added to the water sample (in micrograms)
A(is) = Area of the quantitation ion for the appropriate internal standard
$(\overline{RRF})$ = Average relative response factor for the current initial calibration
V(o) = Original water sample volume (in liters)

Soils:

$$Concentration\ (\mu g/kg) = \frac{A(x) \times I(is)}{A(is) \times \overline{RRF}\ V(g)}$$

Where:

A(x) = Area of the quantitation ion for the compound to be measured

I(is) = Amount of internal standard added to the water sample (in micrograms)

A(is) = Area of the quantitation ion for the appropriate internal standard

$(\overline{RRF})$ = Average relative Response factor from the current initial calibration

V(g) = Original soil sample weight (in grams)

Calculation of the % Error:

$$\% \ Error = \frac{x(i) - x'(i)}{x(i)} \times 100$$

Where:

x(i) = Measured amount of analyte at calibration level *i*, in mass or concentration units
x(i) = True amount of analyte at calibration level *i*, in mass or concentration units

Statistical Information/Method Performance

The LCS/MS and surrogate recoveries and RPD are compared to statistically generated limits for acceptance criteria. The current data is stored in the LIMS under the specific analysis numbers. The historical data for MDLs, MS/MSD, LCS/D, and measurement of uncertainty is reviewed at least annually and updated if necessary. Refer to the QA/QC section of this SOP and the criteria listed throughout this procedure for additional information on the performance of this method.

Quality Assurance/Quality Control

Each extraction batch of no more than 20 samples must contain at least a method blank, laboratory control sample (LCS), and either an unspiked background sample (US), a matrix spike (MS), and a matrix spike duplicate (MSD) or a laboratory control sample/laboratory control sample duplicate (LCS/LCSD). The spiking solution contains all analytes of interest. Additional QC samples may be required to meet project or state requirements.

1. The method blank will be evaluated to determine if any contamination exists. Contamination can occur during the extraction process or during analysis. If target compounds are detected in the method blank above the reporting limit (which may be the MDL or the LOQ depending on each client's reporting requirements), any samples with detections of these same target compounds may need to be re–extracted. Analyze a solvent blank using the same internal standard lot to verify that the analytical system and internal standard solution are free from contamination. Reanalyze the batch blank and if found to be free from contamination, re-inject all samples which contain a positive identification of the same compound. If the batch blank confirms the presence of the contaminant report any sample which

contains a positive detection at a concentration greater than or equal to ten (10) times the concentration in the blank. Any samples which contain a detection less than ten (10) times the concentration in the blank must be re–extracted and reanalyzed. Phthalates may be detected in the method blank and data reported as long as the phthalate detection is < LOQ. A Nonconformance Form *Q-EQA-FRM6869* must be generated and submitted to a supervisor if noncompliant data will be reported.

2. The LCS/LCSD are analyzed to determine the precision of the extraction process. The recoveries of the spiked target compounds should be within the established recovery windows. The established recovery windows are statistically generated and reviewed twice a year. If the recovery for any reported target compound is below QC windows all affected samples will need to be re-extracted. If recoveries are above QC limits, data may be usable if the compounds that recovered above QC limits are not detected in samples. The recoveries for MS/D should be within the established LCS recovery windows.

3. All samples are spiked with 3 surrogate compounds (1-methylnaphthalene-d10, benzo(a)pyrene-d12, and fluoranthene-d10) to evaluate the extraction process. Surrogate windows should be within the established statistical windows. If surrogate recovery does not meet specifications, the samples may need to be re–extracted and reanalyzed. Before re-extraction or re-analysis the analyst should check for calculation errors, instrument performance issues, and matrix interferences.

4. Each sample extract is spiked with an internal standard mix prior to analysis. The internal standard areas from each extract are compared to the internal standard areas from the ICAL. The extract areas must be within –50% to +100% of the ICAL areas. If these criteria are not met, the extract must be reinjected.

5. The lower limit of quantitation (LLOQ) is verified annually through the extraction and analysis of an LCS at 0.5 to 2 times the established LLOQ. The LLOQ is performed on similar analytical instruments such that all are included, at a minimum, within a 3 year time period. Until the laboratory has sufficient data to determine the acceptance limits, the LLOQ criteria is ± 20% of the statistically determined LCS limits.

Table I

DFTPP Key Ion Abundance Criteria (8270C)

Mass	Ion Abundance Criteria
51	30% to 60% of mass 198
68	less than 2% of mass 69
70	less than 2% of mass 69
127	40% to 60% of mass 198
197	less than 1% of mass 198
198	base peak, 100% relative abundance
199	5% to 9% of mass 198
275	10% to 30% of mass 198
365	greater than 1% of mass 198

441	present but less than mass 443
442	greater than 40% of mass 198
443	17% to 23% of mass 442

Table II

DFTPP Key Ion Abundance Criteria (8270D)

Mass	Ion Abundance Criteria
51	10% to 80% of mass 198
68	less than 2% of mass 69
70	less than 2% of mass 69
127	10% to 80% of mass 198
197	less than 2% of mass 198
198	base peak, or >50% mass 442
199	5% to 9% of mass 198
275	10% to 60% of mass 198
365	greater than 1% of mass 198
441	present but less than 24% of mass 442
442	Base peak, or >50% of mass 198
443	15% to 24% of mass 442

Table III

Recommended Minimum Response Factor Criteria for Initial and Continuing Calibration Verification (8270D)

Semivolatile Compounds	Minimum Response Factor (RF)
Naphthalene	0.700
2-Methylnaphthalene	0.400
Dimethylphthalate	0.010
Acenaphthylene	0.900
Acenaphthene	0.900
Dibenzofuran	0.800
Diethylphthalate	0.010

Fluorene	0.900
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Phenanthrene	0.700
Anthracene	0.700
Di-n-butylphthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butylbenzylphthalate	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Benzo(a)anthracene	0.800
Bis(2-ethylhexyl)phthalate	0.010
Di-n-octlyphthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
Benzo(g,h,i)perylene	0.500

Table IV

Semivolatile Internal Standard with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
1,4-Dioxane	Quinoline	Acenaphthene
Benzenethiol	2-Methylnaphthalene	Acenaphthylene
N-Nitrosodimethylamine	Naphthalene	Diethyl phthalate
	Bis(2-chloroethyl)ether	Dimethyl phthalate
	1-Methylnaphthalene	Fluorene
	1-Methylnaphthalene-d10 (surr)	Dibenzofuran

Phenanthrene-d_{10}
Anthracene
Phenanthrene
Di-n-butyl phthalate
Fluoranthene
Hexachlorobenzene
N-Nitrosodiphenylamine
Fluoranthene-d10 (surr)

Chrysene-d_{12}
Pyrene
Benzo(a)anthracene
Bis(2-ethylhexyl) phthalate
Butyl benzyl phthalate
Chrysene

Perylene-d_{12}
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Benzo(a)pyrene-d12 (surr)
Dibenz(a,h)anthracene
Indeno(1,2,3-cd)pyrene
Di-n-octylphthalate
Benzo(e)pyrene
Perylene

(surr) = surrogate

Q-EQA-FRM6869 Nonconformance Form
QA-SOP11178 Demonstrations of Capability
T-SVOA-WI9594 GC/MS Audit Process
T-SVOA-WI9598 GC/MS Preventative and Corrective Maintenance

End of document

Version history

Version	Approval	Revision information
12	08.SEP.2016	
13	15.AUG.2017	
14	21.MAR.2019	

E-13

Sample Preparation of Leachates and Other Wastewater for Analysis of Total Metals by Inductively Coupled Plasma-Mass Spectrometer (ICPMS)
(Rev. 12; 01/19/2015; T-MET-WI11937)

eurofins	Always check on-line for validity.	Level:
Document number: **T-MET-WI11937**	**Sample Preparation of Leachates and Other Wastewater for Analysis of Total Metals by Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)**	**Work Instruction**
Old Reference: **1-P-QM-WI-9015165**		
Version: **12**		Organisation level: **5-Sub-BU**
Approved by: **UBFR** Effective Date **19-JAN-2015**	Document users: **6_EUUSLA_Metals_ICP-MS Prep, 6_EUUSLA_Metals_ICP-MS Prep Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

LIMS ID
Analysis DOD - 6050, 10639

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Revision Log
Reference
Cross Reference
Purpose
Scope
Basic Principles
Reference Modifications
Definitions
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Block Digestion Instructions
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

Revision: 12		**Effective Date:**	This version
Section	**Justification**		**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356		Removed revision logs up to the previous version
Reference	No longer applicable to the procedure		Removed 3010A Modified

Revision: 12	**Effective Date:**	This version
Reference Modifications	No longer applicable to the procedure	Removed 3010A modifications
Procedure A and B	Clarification	Reworded numbers 2, 5 and 6 to clarify steps found in the EPA procedure for 3020A

Revision: 11	**Effective Date:**	Nov 25, 2013
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Cross Reference	Reflect current procedure	Added reference to Analysis #6142, 6123, 6125,…
Sample Collection, Preservation, and Handling	Process change	Changed sample storage temperature from 4° ± 2° C to 0° to 6° C but not frozen, prior to digestion.
Safety Precautions and Waste Handling	No longer used	Deleted text pertaining to Hydrofluoric Acid.
Reagent and Standards	Reflect current procedure	Added text pertaining to preparing solutions using different volumes, is acceptable, if exact ratios are maintained.
Procedure A	Reflect current procedure	Deleted text, in NOTE, pertaining to using a smaller sample aliquot if insufficient sample is submitted. Deleted text, to NOTE, pertaining to samples concentration
Procedure A.1	Reflect current procedure	Added text pertaining to adding spike solution, after the sample has been poured. Added text pertaining to reference to Analysis #6142, 6123, 6125,… for batch quality control requirements.
Procedure B	Reflect current procedure	Deleted text in NOTE pertaining to using a smaller sample aliquot if insufficient sample is submitted. Deleted text to NOTE pertaining to samples concentration
Procedure B.1	Reflect current procedure	Added text pertaining to reference to Analysis #6142, 6123, 6125,… for batch quality control
Block Digestor Instructions	Reflect current procedure	Clarified instruction steps. Deleted text pertaining to the difference between sample temperature and display temperature.
Quality Assurance/Quality Control	Reflect current procedure	Added reference to Analysis #6142, 6123, 6125,… for batch quality control requirements.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3020A, July 1992.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …	Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A (aqueous, solid, tissue) and EPA 200.8 (aqueous)
1–P–QM–FOR–9009182	Working Instructions for Prep Solutions and Standards
1–P–QM–QMA–9015390	Demonstrations of Capability

Purpose

This digestion procedure is used to prepare leachate and other wastewater samples for measurement of total metals by inductively coupled plasma-mass spectrometer (ICP–MS) following SW 846 protocol.

Scope

This acid digestion procedure is used by the Metals Department of the Environmental Sciences Division to prepare leachate, wastewater, surface water, and groundwater samples for measurement of total recoverable metals by inductively coupled plasma-Mass Spectrometer (ICP-MS) following SW-846.

Basic Principles

A mixture of nitric acid and the sample is refluxed in a covered beaker/digestion vessel at low volume to dissolve metals. It is cooled and brought up to volume with reagent water.

Reference Modifications

1. A 50–mL sample aliquot and final volume is used instead of 100–mL to improve digestion throughput, conserve sample usage, and limit waste generation. Because all reagents are also adjusted so that concentrations are equivalent to a 100–mL aliquot, there is no impact on the data.

2. Ribbed watch glasses are not used; samples are evaporated without watch glasses in nonmetallic hoods to speed evaporation. No contamination trends have been observed in prep blanks evaporated without watch glasses.

Definitions

1. ACS – American Chemical Society

2. ASTM – American Society of Testing and Materials

3. D – Sample Duplicate

4. DOC – Demonstration of Capability

5. IDOC – Initial Demonstration of Capability

6. LCS/LCSD – Laboratory Control Sample/ Laboratory Control Sample Duplicate

7. LCSW – Laboratory Control Sample Water

8. LLENS – the computer program that integrates a PC with an analytical balance to collect data directly from the balance. The program organizes the data and transmits the readings to the LIMS.

9. LIMS – Laboratory Information Management Systems

10. LLI Sample ID – unique 7-digit number assigned to a client sample.

11. LOQ – Limit of Quantitation

12. MDL – Method Detection Limit

13. MS (R) – Matrix Spike

14. MSD (M) – Matrix spike duplicate

15. PB/PBW – Preparation Blank/ Preparation Blank Water

16. QC – Quality Control

17. Method Blank – equivalent to a Preparation Blank. A designated sample designed to monitor for sample contamination during the analysis process. A volume of reagent laboratory water is typically used to monitor water sample analysis, while solids blanks consist of a purified solid matrix

or just the reagents used in the test. The blank demonstrates that no artifacts were introduced during the analysis process.

18. SOP – Standard Operating Procedure

19. SPLP – Synthetic Precipitation Leaching Procedure

20. STLC – Soluble Threshold Limit Concentration

21. TCLP – Toxicity Characteristic Leaching Procedure

22. U or US – unspiked background sample

Interferences
Not applicable to this procedure.

Safety Precautions and Waste Handling
All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

> **Nitric acid (HNO_3)** – This acid can cause skin burns. Add nitric acid to samples in a hood or use the designated dispensing equipment to avoid exposure to toxic fumes.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds.

Samples that contain dust may be hazardous. Open in a fume hood.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A , soda ash or equivalent, to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf. Soda ash is located in the stairwell adjacent to the prep room.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability for this or an equivalent procedure.

Initially, each employee performing this digestion procedure must work with an experienced employee for a period of time until they can independently set up batches and perform the necessary steps outlined in this procedure. Proficiency is measured through documentation of the critical steps in this procedure, over checking of data as well as an IDOC.

The IDOC and the DOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Refer to 1−P−QM−QMA−9015390, for specific requirements. A DOC is performed annually and is maintained in the analyst's training records.

Sample Collection, Preservation, and Handling

Samples are collected in plastic containers and preserved to a pH of <2 with HNO_3. (Samples to be analyzed for soluble metals requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45−micron filter within 5 days of receipt and then preserved.) The pH is checked upon receipt and adjusted as necessary by Sample Support; samples that are pH adjusted at the lab must not be digested for a minimum of 24 hours. If samples fail to maintain a pH of <2 the Client Service Representative is notified for further direction. Samples are stored at 0° - 6°C, but not frozen, prior to digestion. Samples must be digested within 6 months of collection. Digested samples are stored in plastic at room temperature and have a 6 month holding time.

Apparatus and Equipment

1. Polypropylene containers (digestion vessels) – certified clean and Class A equivalent
2. Watch glasses or reflux caps
3. 50−mL graduated cylinders or other appropriate graduated cylinders if necessary
4. 50−mL volumetric flasks or other appropriate Class A volumetric flasks if necessary
5. 250-mL beakers or other appropriate beakers
6. Hotblocks or hot plates, adjustable and capable of maintaining a temperature of 90° to 95°C

Reagents and Standards

For reagent preparation, shelf life, and storage conditions, see Form 1−P−QM−FOR−9009182. .

Nitric acid, HNO_3 – Fisher, Trace Metal Grade, or equivalent. Store at room temperature and re-evaluate annually.

NOTE: It is acceptable to prepare solutions using multiples of indicated volumes if exact ratios are maintained.

Calibration

Not applicable to this method.

Procedure

This SOP has been set up to outline the procedures for both hotblock and hot plate digestions (see below). Choose the procedure that corresponds to the sample heating technique being used for sample digestion.

A. Hotblock

NOTE: When insoluble matter is present in the digested sample, allow it to settle by gravity or filter prior to introduction to the instrument. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45−micron filter paper. Adjust the filtered sample to pH <2 with nitric acid preserving solution. Measure the volume of sample, as stated in this procedure, and digest as normal. The prep blank and spiked LCS must also be prepared with filtered water.

NOTE: If the sample contains high solids, use a smaller aliquot of the sample and bring sample to final volume as stated in this procedure. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

1. Shake sample well. Transfer 50 mL of well mixed sample to a 68−mL digestion vessel. After the sample has been poured, add the spiking solution. For sample batch spiking procedures see form 1−P−QM−FOR−9009182. For sample batch quality control requirements see Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …

NOTE: For leachate samples, use the appropriate extraction fluid for the PBW and LCS. The extraction fluids are as follows: TCLP, SPLP, STLC, ASTM, Filtration, and Elutriate.

2. Add 1.5 mL of HNO_3. Place the vessel in the hotblock at 90° to 95°C, and cautiously evaporate to low volume (about 5 mL), making certain that the sample does not boil and that no portion of the bottom of the digestion vessel is allowed to go dry.

3. Cool the digestion vessel and add another 1.5−mL portion of HNO_3.

4. Cover the digestion vessel with a reflux cap and return to the hotblock. Increase the temperature of the hotblock so that gentle reflux action occurs.

NOTE: If a sample is allowed to go to dryness, low recoveries result. If this occurs, discard the sample and re-prepare in a new batch.

5. Continue heating (refluxing), adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

6. When digestion is complete, uncover the digestion vessel and evaporate to low volume (about 3 mL). Do not allow any portion of the bottom of the digestion vessel to go dry.

7. Remove the digestion vessel and add approximately 5 mL of reagent water, mix, and continue warming for 10 to 15 minutes to allow additional solubilization of any residue to occur.

8. Allow to cool. Adjust volume to the 50−mL mark on the digestion vessel with reagent water and mix. Seal vessel with screw cap.

9. The sample is now ready for analysis.

B. Hot Plates

NOTE: When insoluble matter is present in the digested sample, allow it to settle by gravity or filter prior to introduction to the instrument. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45−micron filter paper. Adjust the filtered sample to pH <2 with nitric acid preserving solution. Measure the volume of sample, as stated in this procedure, and digest as normal. The prep blank and spiked LCS must also be prepared with filtered reagent water.

NOTE: If the sample contains high solids, use a smaller aliquot of the sample and bring sample to final volume as stated in this procedure. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

1. Shake sample well. Use a 50 mL graduated cylinder to transfer 50 mL of well mixed sample into a 250 mL beaker. After the sample has been poured, add the spiking solution. For sample batch spiking procedures see form 1−P−QM−FOR−9009182. For sample batch quality control requirements see Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …

NOTE: For leachate samples, use the appropriate extraction fluid for the PBW and LCS. The extraction fluid are as follows: TCLP, SPLP, STLC, ASTM, Filtration, and Elutriate.

2. Add 1.5 mL of HNO_3. Place the beaker on a hot plate and cautiously evaporate to low volume (about 5 mL), making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry.

3. Cool the beaker and add another 1.5–mL portion of HNO_3.

4. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that gentle reflux action occurs.

NOTE: If a sample is allowed to go to dryness, low recoveries result. If this occurs, discard the sample and reprepare in a new batch.

5. Continue heating (refluxing), adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

6. When digestion is complete, uncover the beaker and evaporate to low volume (about 3 mL). Do not allow any portion of the bottom of the beaker to go dry.

7. Remove the beaker and add approximately 5 mL of reagent water, mix, and continue warming the beaker for 10 to 15 minutes to allow additional solubilization of any residue to occur.

8. Allow to cool. Transfer the solution to a 50–mL volumetric flask. Adjust volume to the 50 mL mark with reagent water and mix.

9. Transfer to a polypropylene bottle.

10. The sample is now ready for analysis.

Block Digestion Instructions

1. Turn block digestor on by pressing rocker switch located on the cord.

2. Wait about 8 seconds until controller display indicates current block temperature.

3. PRESS and hold STAR (*) key.

4. The display shows the Set Point Temperature.

5. The digits can be changed to the desired value by pressing the up and down arrow keys while holding the (*) key.

6. Confirm Control Point temperature is set to the block temperature that provides 90° to 95°C.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used for digestion. If necessary, adjust Control Point temperature to the proper setting.

NOTE: Polypropylene containers must not be heated above 130°C.

Calculations

Not applicable to this procedure.

Statistical Information/Method Performance

Not applicable to this method. See analysis procedure.

Quality Assurance/Quality Control

A method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample must be performed with every digestion batch (20 samples or less). Each piece of batch QC is digested following the procedure in this SOP.

For sample batch quality control requirements see Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …

End of document

Version history

Version	Approval	Revision information
12	19.JAN.2015	

E-14

Metals by Inductively Coupled Plasma Mass Spectrometry for SW846 Methods 6020/6020A/6020B (aqueous, solid, tissue) and EPA 200.8 (aqueous)
(Rev. 9; 03/21/2019; T-MET-WI11933)

eurofins	Always check on-line for validity.	Level:
Document number: **T-MET-WI11933**	**Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A/6020B(aqueous, solid, tissue) and EPA 200.8 (aqueous)**	**Work Instruction**
Old Reference: **1-P-QM-WI-9018443**		
Version: **9**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **21-MAR-2019**	Document users: **6_EUUSLA_Metals_ICP-MS Analysis, 6_EUUSLA_Metals_ICP-MS Analysis Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

LIMS ID

Water Analysis # 6027, 6043, 6036, 6023, 6040, 6029, 6047, 6048, 6031, 6037, 6034, 6032, 6039, 6033, 6049, 6025, 6041, 13171, 6044, 6038, 6042, 6028, 6046, 6024, 6026, 6045, 6035, 13501 Solid Analysis # 6127, 6143, 6136, 6123, 6140, 6129, 6147, 6148, 6131, 6137, 6134, 6132, 6139, 6133, 6149, 6125, 6141, 13172, 6144, 6138, 6142, 6128, 6146, 6124, 6126, 6145, 6135, 13502

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Table II - ICP-MS Metals by EPA 6020/6020A/6020B

Revision Log

Revision: 9	**Effective Date:**	This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version.
Scope	Redundant Information	Deleted reference to elements list since this is already referenced in the procedure elsewhere.
Routine Methods	Clarification	Changed wording to include all ICP-MS, not specifying individual instruments.
Basic Principles	Addition	Added instrument names and clarification about instrument analysis modes for gas and no gas modes.
Interferences	Clarification	Added that rinsing is done with an appropriate rinse solution
Safety Precautions and Waste Handling	Added/Deleted	Clarified information about which acids are used and how to use them safely, removed information about concentrated HF acid that is not used in this analysis. Only trace levels are used. Adjusted information according to trace use. Corrected the information on response to acid spills to current practice and EHS policies.
Personnel Training and Qualifications	Readability/Clarification	Added reference to DOC document *QA-SOP11178* and clarified data transfer for iDOCs.
Sample Collection, Preservation, and Handling	Readability/Clarification	Grammatical errors corrected.
Apparatus and Equipment	Addition	Added information regarding ICP MS equipment changes and the addition of the Agilent 7900.
Reagents and Standards	Readability/Clarification	
Calibration	Added/Deleted	Removed redundant information referencing forms in the previous section. Clarified warm up time requirements and when full tuning and optimization is done or needed. Clarified when a single point and multi-point calibration is done.
Procedure	Added/Deleted	Updated procedure for current practices and current equipment/software changes and added clarification for procedure steps when preparing a batch for analysis.

Revision	8	**Effective Date:**	25-SEP-2018
Section	**Justification**		**Changes**
Revision Log	Formatting requirement		Removed revision logs up to the previous version.
Table of Contents	Removed		Not needed.
Purpose	Redundant information		Deleted Section
Routine Methods	Not required		Removed Li, P, Zr, Rb (10803, 10804, 10805, 10806, 10807, 10808, 13171, 13172)

Revision	8	**Effective Date:**	25-SEP-2018
Instrument Operations	Readability/Clarity		Moved to working instructions form.
Instrument Maintenance	Readability/Clarity		Moved to working instructions form.
Throughout Document	ELAN 9000 Not required Added new Agilent 7900		Information concerning the Perkin Elmer ELAN 9000 is no longer required. Added new Agilent 7900.
Cross Reference, Apparatus and Equipment	Addition		Added new forms numbers for instrument operations, maintenance.
Scope	Correction		Added matrices.
Sample Collection	Correction		Changed 180 day hold time to 6 months to analysis and clarified the storage requirements. Removed extraneous information located in other SOPs.
Calibration	Addition		Added information on single point and multipoint calibration. Added Form references for instrument operations.
Procedure B. 6.	Addition		Added Laboratory Control Sample as a requirement to be filtered as well as the Prep Blank if samples on the batch are filtered.
Table I	Addition		Added Prep Blank criteria of <2.2x MDLfor 200.8
Table I and II	Addition		Added calibration frequency as daily.

Reference

1. Method 200.8 (rev 5.4), Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry, USEPA 600/R-94/111 May 1994.

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020, September 1994.

3. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020A, February 2007.

4. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020B, Rev. 2, July 2014.

5. Agilent 7500 Series ICP-MS ChemStation (G1834B) Operator's Manual, Hardware Manual, Tuning & Application Handbook, and Maintenance Video (DVD).

6. ESI SC Manual.

7. Agilent 7700 and 7900 Series ICP-MS MassHunter Workstation Guide and Hardware Maintenance Manual.

8. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Q-EQA-FRM6869	Nonconformance Form
T-MET-WI12065	Working Instructions for Preparation of ICP-MS Solutions and Standards
S-SS-WI10695	Liquid Sample Preservation
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11901	Instrument and Equipment Maintenance and Calibration
T-MET-WI18026	Instrument Operations for Agilent 7500
T-MET-WI18027	Instrument Operations for Agilent 7700
T-MET-WI18028	Instrument Maintenance for Agilent 7500
T-MET-WI18029	Instrument Maintenance for Agilent 7700
T-MET-WI21589	Instrument Operations for Agilent 7900
T-MET-WI21590	Instrument Maintenance for Agilent 7900
T-MET-FRM8822	ICPMS Analysis

Scope

This procedure applies to analyses performed at Eurofins Lancaster Laboratories Environmental (ELLE) using ICP-MS for identification and quantitation of metallic constituents.

Aqueous undigested samples and digestates (aqueous and solid matrices) are analyzed via this SOP.

Limits of Quantitation (LOQs), Method Detection Limits (MDLs) and linear ranges are maintained in the LIMS. These are subject to change without notification.

Routine Methods

Elements routinely analyzed on ICP-MS are outlined in *T-MET-FRM8822* and include the following:

Analyte	Mass	Soil Analysis LIMS #	Water Analysis LIMS #
Be	9	6127	6027
B	11	10801	10802
Na	23	6143	6043
Mg	24	6136	6036
Al	27	6123	6023
K	39	6140	6040
Ca	44	6129	6029

Ti	47	6147	6047
V	51	6148	6048
Cr	52	6131	6031
Mn	55	6137	6037
Fe	57	6134	6034
Co	59	6132	6032
Ni	60	6139	6039
Cu	63	6133	6033
Zn	66	6149	6049
As	75	6125	6025
Se*	78	6141	6041
Se**	82	6141	6041
Sr	88	6144	6044
Mo	98	6138	6038
Ag	107	6142	6042
Cd	111	6128	6028
Sn	120	6146	6046
Sb	121	6124	6024
Ba	137	6126	6026
Tl	203	6145	6045
Pb 206	206	6135	6035
Pb 207	207	6135	6035
Pb 208***	208	6135	6035
U	238	13502	13501

*Se 78 is the mass reported from collision cell ICPMS only
**Se 82 is the mass reported from non-collision cell ICPMS only
***For Pb masses 206, 207 and 208 are summed for calibration and analysis

Basic Principles

ICP-MS is an analytical instrument that uses the energy of inductively coupled plasma to generate ions to be analyzed in the mass spectrometer.

The instruments use a discrete sampling system that first loads the sample into a Teflon sample loop. The sample is then pushed by a carrier solution using a computer controlled peristaltic pump that delivers the sample from the loop into the nebulizer attached to an electronically cooled cyclonic spray chamber.

The Agilent 7500 (E03), 7700 (E05) and 7900 (E07) can analyze samples in 3 different modes of operation, although typically analysis is done in 3 mode or He mode only:

1. Mode 1 – no gas. This mode does not use a collision or reaction gas. Elements with a low atomic mass or that have no interferences can be analyzed using the "no gas" mode.

2. Mode 2 – Helium (He) mode. This mode uses He as a collision gas for reliable, predictable removal of unknown matrix interferences. No new interferences are formed in the cell, and no analytes are lost by a reaction.

3. Mode 3 – Hydrogen (H2) mode. This mode uses H2 as a reaction gas, since it reacts quickly and efficiently with the Argon-based interfering species, but reacts slowly or not at all with the analyte(s) of interest. In this mode, interferences can be reduced to the level of baseline noise, allowing lower detection limits to be achieved for some difficult elements.

Interferences

ICP-MS interferences include isobaric elemental interferences and polyatomic ion interferences derived from the plasma gas, reagents, and/or sample matrix. The Agilent 7500, 7700 and 7900 utilize helium as an inert collision gas to reduce or eliminate many types of polyatomic ion interferences.

Physical interferences caused by the change in sample matrix affecting sample transport and/or nebulization must be compensated for using internal standardization.

Memory interference is the contribution of analyte signal from a previous sample onto the next sample analysis. Adequate rinse time with an appropriate rinse solution of the sample introduction system overcomes any memory interference.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.
See the *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Hydrochloric acid (HCl) – This acid can cause skin burns. Only mix with approved solutions. Always use this chemical in a fume hood.

Hydrofluoric acid (HF) – This acid is very toxic and absorbs through the skin without pain. This acid is found at trace levels in standard solutions.
Hydrogen Peroxide 30% (H2O2) **-** This oxidizer can cause skin burns. Always use in a fume hood.

Nitric acid (HNO3) – This acid can cause skin burns and burning of the respiratory tract. Add nitric acid to samples in a fume hood to avoid exposure to toxic fumes.

When diluting strong acids, never add water to acid; always add acid to water.
Store concentrated acids in the prep room acid cabinets. Only acids are to be stored in these cabinets. Flammable solvents are stored in flammable liquid storage cabinets. Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Large spills are cleaned by HSE. For large spills of concentrated acid, immediately evacuate the area and dial 1-1-1. HSE will use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A, soda ash or equivalent, to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf. Soda ash is located in the stairwell adjacent to the prep room.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the sequence editor to set up the run, perform calculations, interpret raw data, and enter/transfer data into the LIMS. Proficiency is measured through documented audits of the tasks listed, and checking of data as well as an IDOC (Initial Demonstration of Capability) and continued DOCs (Demonstration of Capability) as defined in *QA-SOP11178*.

Sample Collection, Preservation, and Handling

Aqueous undigested samples, aqueous digestates, and solid digestates are analyzed via this SOP. Samples must be analyzed within 6 months of collection for all methods in this SOP.

Aqueous samples are collected in plastic or glass containers. Drinking Water samples are collected in 1-L plastic or glass containers. Aqueous samples are preserved with nitric acid with a pH of < 2 and stored at 0° to 6°C, not frozen.

Aqueous digestates are digested per the method in SOP *T-MET-WI11939* (aqueous samples). Aqueous samples are stored in 50 ml plastic vials at room temperature.

Solid samples are collected in glass containers and stored at 0° to 6°C, not frozen. Refer to the protocols in the digestion SOP T-MET-WI8636. Solid samples are stored in 100 ml plastic vials at room temperature.

Store standards and digestates separately.

The general practice in the metals group is to discard the digestions after all the required metals from a batch of samples have been analyzed and verified in the LIMS. Samples which require the digestate to be held for long term storage are periodically evaluated for discard.

Apparatus and Equipment

A. The following is a list of the hardware used in the Agilent 7500, 7700 and 7900 ICP-MS systems.

1. Inductively Coupled Plasma Mass Spectrometers (ICP MS)

   a. Agilent 7500ce - The sample introduction system consists of an ESI FAST discrete sampling system, Teflon nebulizer and electronically cooled spray chamber attached to a concentric quartz tube plasma torch.

   b. The Agilent 7700 and 7900 - The sample introduction system consists of an ESI FAST discrete sampling system, glass concentric or Teflon nebulizer and electronically cooled spray chamber attached to a concentric quartz tube plasma torch.

2. Autosampler

   a. The Agilent 7500 and 7700 systems use an ESI SC-8 FAST autosampler. The Agilent 7900 system uses an ESI SC-14 FAST autosampler. The autosampler parameters for each automated run are entered into the Edit Sample Log Table interface editor in the ChemStation software (7500) or in the Sample List in MassHunter software (7700 and 7900).

3. Coolflow

   a. The Agilent 7500 and 7900 G3292A Recirculating Chillers are set up to deliver cooling liquid to the ICP-MS at a regulated pressure of 60 psi.

   b. The Agilent 7700 G1879B Heat Exchanger is set up to deliver cooling liquid to the ICP-MS at a regulated pressure of 30 psi.

4. Computer

   a. The Agilent 7500ce is controlled by a Windows-based IBM compatible PC with ICP-MS ChemStation software installed.

b. The Agilent 7700 and 7900 is controlled by a Windows-based IBM compatible PC with MassHunter software installed.

5. Vacuum Pumps

a. The Agilent 7500ce, 7700 and 7900 both have a 3-stage vacuum system. A rotary pump evacuates the interface chamber; a turbomolecular pump evacuates the ion lens chamber; and a turbomolecular pump evacuates the analyzer chamber.

B. The following is a list of the apparatus necessary for the setup of an ICP-MS run for analysis:

1. ICP-MS run cover sheets. Cover sheets are generated using the IDAT Sequence Editor program in LIMS.

2. Test tube racks

3. 17 ´ 100-mm polystyrene tubes

4. Graduated 15-mL polypropylene tubes and caps

5. 50-mL polypropylene screw cap tubes

6. FilterMate 2 µm filters and plungers

7. 1 ´ 100 10-mL sterile disposable syringes

8. 25-mm syringe filters, PTFE, 0.45 µm

9. 30-mL polypropylene medicine cups

10. Adjustable electronic hand-held pipettes (10 - 5000 µL) - Fisherbrand® or equivalent.

NOTE: For routine operation, calibration, and maintenance of adjustable electronic hand-held pipettes, see *QA-SOP11880*.

11. Fixed volume hand-held pipettes (25 – 2000 µL) - Eppendorf or equivalent.

NOTE: For routine operation, calibration, and maintenance of fixed volume hand-held pipettes, see *QA-SOP11880*

Reagents and Standards

Refer to Form *T-MET-WI12065* for reagent and standard information and the preparation of the following standards and solutions

1. ICP-MS Calibration Standards and ICP-MS LOQ Check Standard Solution (LLC)
2. ICP-MS Initial and Continuing Calibration Verification Standards
3. Interference Check Solutions
4. Rinse/Carrier, Tuning, and Calibration Solutions
5. Internal Standard Solution
6. PDS Solutions
7. General Acids and Chemicals

Calibration

Before calibration is performed, allow the instrument to warm up for the recommended time, usually at least 15 minutes, if the plasma has been off for 15 minutes or less, or at least 30 minutes if the plasma has been off for more than 15 minutes. Daily tuning requirements must be performed prior to calibration of the instrument and after warm up occurs. Refer to forms *T-MET-WI18026*, *T-MET-WI18027* and *T-MET-WI21589* for detailed tuning and calibration information. Full Tuning and Optimization is done as needed, or when required by the Group Leader.

Calibration is performed daily. Calibrations consist of a blank and one to three standards, followed by analysis of an ICV, ICB, CCV, LLC, ICSA/ICSAB, CCV and CCB.

A single point calibration, consisting of a blank and one standard, is used for all analysis except samples requiring a multi-point calibration (either MA MCP or CT RCP protocol or a lab note indicating a multi-point calibration is needed). For samples requiring a multi-point calibration the calibration consists of a blank, "S1", "S2" and "S3" standards. The concentrations of the calibration standards are defined in *T-MET-WI12065*.

For acceptance criteria and corrective action see Table I and Table II.

Procedure

A. Setting up an ICP-MS run

The procedure is the same for the Agilent 7500 (E03), 7700 (E05), and 7900 (E07) instruments.

1. Determine the batches to be analyzed and determine any special requirements by viewing lab notes and/or project notes that are with the batch paperwork.

2. Open the Sequence Editor program from the IDAT menu in eLIMS.

3. Select "ICP-MS" in the Instrument Method drop down list.

4. Select the appropriate digest type and load the list of batches.

Note: Use the checkbox "Show scheduled batches" if you need a batch that has been written up previously. This will show all batches that have not been written up before and batches previously written up.

5. Select the appropriate batch and then the template. Add the batch to the template using the "Add Batch" button.

6. Review the information uploaded to the Sequence Editor program.

   a. Information automatically included as required by template:

      (1) PBW – Prep blank (water)
      (2) LCSW – Laboratory control sample (water)
      (3) LCSDW – Laboratory control sample duplicate (water)
      (4) PBS – Prep blank (solid)
      (5) LCSS – Laboratory control sample (solid)
      (6) LCSDS – Laboratory control sample duplicate (solid)
      (7) Lancaster Laboratories' sample number
      (8) CCV – Continuing calibration verification
      (9) CCB – Continuing calibration blank
      (10) LLC – Low level check
      (11) ICSA – Interference check standard – A
      (12) ICSAB – Interference check standard - AB
      (13) S0 – Calibration blank
      (14) S1 – Calibration standard 1
      (15) S2 – Calibration standard 2 - Multi-point only
      (16) S3 – Calibration standard 3 - Multi-point only
      (17) CCS – Carryover Control Standard - rinse
      (18) Batch No. – The batch number of the sample.

   b. IV, FV and SDG are not included on the cover sheet but are pulled from LIMS during import.

      Initial volume (IV) – The sample aliquot digested.
      Final volume (FV) – The final sample volume after digestion

      SDG – Sample delivery group number for data package samples.

c. Dilution factor (DF) – The dilution factor of the sample prepared at the time of analysis, needed to bring the sample into the linear range of the instrument, to negate a matrix effect, or for serial dilutions. If dilutions other than the default dilution are required, the analyst must change the DF for affected tubes prior to saving the sequence. The sample may be added multiple times for multiple dilutions if required.

d. Protocol – The protocol used to review the data for specific method requirements in the IDAT database. Each template in Sequence Editor has a default protocol. If samples require a different protocol it must be changed for the appropriate tubes prior to saving the sequence.

e. Comments – Any description of the sample (from prep logs), status of the sample (i.e., RUSH, Promised) and due date are recorded here. The list of elements needed for the run is automatically inserted on the cover sheet by the Sequence Editor Program. Additional comments may be recorded under the Elements column if additional space is needed.

f. Lot numbers – Lot numbers for filters, test tubes, and standards must be recorded by the analyst at the start of the analytical run.

g. Run Name – The Run Name is recorded by the analyst at the start of the analytical run.

7. When setting up a run using the Sequence Editor Program, Batch QC (i.e., PB, LCS, background, duplicate, matrix spike, matrix spike duplicate, post-digest spike, and serial dilution) are automatically placed in a block of ten or fewer samples. If there is an LCS and LCSD, they are automatically placed one after the other.

a. ICV/ICB must be analyzed immediately after the calibration curve.

b. CCV/CCB must be run after every ten analytical samples.

c. LLC, ICSA, ICSAB, CCV, and CCB immediately follow the ICV/ICB. LLC, ICSA, ICSAB, CCV and CCB conclude each run per client requirement. DOD does not require an ending ICSA/ICSAB

d. Any deviations from protocol must be noted in the Comments Section of the cover page.

e. Verify that all information has been entered correctly.

f. Save the file. This automatically generates the cover sheet and opens the cover sheet in a Word document.

g. Add batch location and any comments to the cover sheet.

h. Print the cover sheet.

B. Pouring an ICP-MS run (Waters only in 50 mL digestion tubes):

The procedure is the same for the Agilent 7500 (E03), 7700 (E05) and 7900 (E07) instruments.

It is important to minimize any chance of contamination, to both yourself and the samples. Keep your hands and the work area clean at all times. Wear appropriate PPE at all times to prevent exposure to chemicals and samples.

NOTE: See Form *T-MET-WI12065* for standards and solutions used during the analytical run.

1. Choose the appropriate run sheet.

NOTE: When retrieving the batch from the shelf for the first time, record the batch location on the cap of the prep blank in black sharpie. This saves time when returning the batch to the proper location after analysis and shows that the batch has been poured before and verified to be in the correct order. To prevent more than one batch from being assigned to the same location, the digestion trays all have the location written on the foam holder.

2. Verify that each digestion tube is in the correct order according to the cover sheet. If anything is missing, correct the cover sheet and notify PREP or the Group Lead to correct LLENS and put the sample back in for digestion. If the order is incorrect, reorder the sample digestion tubes to match the paperwork.

3. For batches prepared in hot blocks in 50 mL digestion tubes:

   a. The only tubes that typically need to be poured for the initial run are the post digestion spike (PDS, or UP) and serial dilution (UL) samples. These are prepared using the background sample (U) or the duplicate background sample (D) when limited sample volume is available.

   b. All other samples remain in the 50 mL digestion tubes for analysis.

4. Samples that need to be poured into test tubes:

   a. Obtain the appropriate number of tubes.

   b. Write the last four digits of the sample number and the dilution factor if greater than 1 on each tube. If the PBW/PBS or LCS/LCSW must be in tubes, write PBW/PBS/LCS/LCSW and the batch location on the tubes.

   c. Place tubes in test tube racks in the same order as the run on the cover sheet.

   d. The analyst must ensure that tubes are labeled correctly, that dilutions are performed accurately, and that the dilutions are recorded on the run cover sheet.

5. Post-digest spike (PDS):

a. A PDS is required for each batch (sample volume permitting).

b. A PDS is prepared by pipetting 0.2 mL of the appropriate PDS solution into a 14 mL graduated test tube, and bringing it to a volume of 10 mL with the background sample, "U". If the PDS must be diluted, pipette 0.2 mL of the PDS solution into a 14 mL graduated test tube, add the appropriate amount of background sample, "U", and dilute to 10 mL using matrix rinse. If there is limited volume for the background sample, "U", then the "D" may be used for dilutions of the PDS or UL.

c. Record the lot number of PDS solution used in the comments column of the ICP-MS run sheet.

6. Serial dilution SD or (UL)

a. Prepare the SD (UL) by diluting the background sample at a dilution that is equal to 5× the dilution factor of the PDS (UP) sample (i.e. if PDS (UP) = DF1, SD (UL) must = DF5; if PDS (UP) = DF5, SD (UL) must = DF25).

7. Filtering samples

a. Samples that are cloudy or have particulate suspended in solution must be filtered prior to analysis to prevent clogging of the sample introduction system, which causes run failures and instrument down time to correct the problem.

b. Samples that are digested using the 50 mL hot block tubes need to be filtered using the FilterMate filtration devices and plunger.

c. Attach a filtration device to the plunger and very carefully insert the filter into the hot block tube until the filter reaches the bottom of the digestion tube.

d. Remove and discard the plunger.

e. An alternative for samples in any type of vessel is to filter using a 10-mL sterile disposable syringe fitted with a 0.45 µm PTFE syringe filter.

f. If any samples are filtered, the PB and LCS must also be filtered. If any QC sample is filtered (U, D, R or M), then all QC including the PB and LCS must be filtered.

g. Document all filtrations on the run cover sheet.

NOTE: It is not necessary to filter all samples (in hot block tubes only) that contain particulate, as long as all of the particulate is settled to the bottom of the tube below the 5 mL mark in 50 mL digestion vessels. The sampling depth of the autosampler probe is set such that the tip of the probe is approximately 1 cm above the bottom of the digestion tube.

8. Verify that all samples in the hot block tray are in the correct position and/or pour each sample (or sample filtrate) into the appropriate tube. Usually, the order of the batch QC is PB, LCS, (LCSD), Bkg, PDS, DUP, MS, (MSD), and SD. Most importantly, the actual positions of the samples in the tray must match the autosampler table used for analysis.

NOTE: Immediately prior to beginning analysis, the analyst must visually verify that each sample is in the position indicated in the autosampler table used for the run.

9. If not being analyzed immediately, cover any poured tubes with plastic wrap to prevent contamination of the samples (hotblock tubes and dilutions prepared in graduated test tubes must be capped tightly except during pouring and/or analysis).

10. Return samples to sample storage, being sure to place the batch back into the location recorded on the batch sheet.

11. Record the following on the first page of the ICP-MS run sheet under "Poured By": initials, employee number, and the date.

12. NOTES:

    a. A PDS (UP) and a SD (UL) are performed on one sample in each digestion batch. Typically, the background sample is chosen. If the batch QC is split between two samples, the PDS is performed on the background sample accompanied by a matrix spike; the SD is performed on the background sample accompanied by a matrix duplicate. If sample volume is limited, it is acceptable to use the duplicate for the PDS and SD.

    b. Batches with only field blanks or equipment blanks do not need a PDS or a SD.

    c. Air filter batches need only a SD on one sample in the batch (a PDS is not required).

    d. “As Received” samples are analyzed with a blank and LCS, LCSD (prepared by the analyst). These “batches” are assigned a batch number by the analyst and recorded into the departmental LLENS system and is uploaded to LIMS.

    e. Documentation is of utmost importance. Verify all entries.

Calculations

1. Final Result

   a. Water sample

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

b. Solid sample (mg/kg)

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\%\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{\left(\sum X^2 - \frac{(\sum X)^2}{N}\right)\left(\sum Y^2 - \frac{(\sum Y)^2}{N}\right)}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\%\ Difference = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Methods of standard additions (MSA)
Take either 4 identical aliquots (for 3 point MSA) or 2 identical aliquots (for one point MSA) of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution (for 3 point MSA) and spike the other aliquot at approximately the indigenous concentration of the sample (for one point MSA). Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots or 2 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y- intercepts. The x- intercept is the result.

$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i)/n}{\sum x_i^2 - (\sum x_i)^2/n}$$

$$\text{Y-Intercept} = b = \bar{y} - m\bar{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum\{(x_i - \bar{x})(y_i - \bar{y})\}}{\sqrt{\left[\sum (x_i - \bar{x})^2\right]\left[\sum (y_i - \bar{y})^2\right]}}$$

The correlation coefficient (r) for the least squares fit must be ³0.995. If the r value is <0.995, the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is <0.990, the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

7. Average, Standard Deviation and Relative Standard Deviation

The average result, $\bar{x}$, is calculated by summing the individual results and dividing this sum by the number (n) of individual values:

$$\bar{x} = \frac{x_1 + x_2 + x_3 + x_4 + \ldots x_n}{n}$$

Standard deviation is a measure of how precise the average is or how well the individual numbers agree with each other.

$$\text{Standard deviation, } S = \sqrt{\frac{(x_1-\bar{x})^2+(x_2-\bar{x})^2+(x_3-\bar{x})^2+\ldots+(x_n-\bar{x})^2}{n-1}}$$

Relative standard deviation (RSD) is expressed in percent and is obtained by multiplying the standard deviation by 100 and dividing this product by the average.

$$\text{Relative standard deviation, RSD} = \frac{100\times S}{\bar{x}}$$

Statistical Information/Method Performance

Generate MDLs and LOQs according to *QA-SOP11892*.

The department supervisor maintains annual study data. The department supervisor requests that a Quality Assurance Specialist update to the LIMS as needed. Update the department database via a download from the LIMS.

Refer to *QA-SOP11896* for statistical information.

Quality Assurance/Quality Control

A. For 6020, 6020A and 6020B, each digestion batch (up to 20 samples) must contain a method blank, LCS, and either an US, D, MS, MSD or an LCS/LCSD.

B. For 200.8, each digestion batch (up to 10 samples) must contain a method blank, LCS, and either an US, D, MS or an LCS/LCSD.

C. QC limits for MS/MSD, and LCS/LCSD are established through statistical analysis of historical data.

1. The limits are maintained in the LIMS for the relevant analysis numbers.

2. The limits are evaluated every 6 months and updated as needed.

3. The limits are subject to change without notification.

D. Batch Quality Control

1. For the preparation and concentrations of Batch Quality Control see *T-MET-WI12065*.

2. For the frequency, acceptance criteria and corrective action see Tables I and II.

E. Raw data quality checks

1. Make sure that the run is correctly labeled, and dated and that the corresponding cover sheet is attached to the run.

2. Verify that the appropriate Tuning Report is with the run.

3. For run and batch QC/Calibration frequency, acceptance criteria and corrective action, see Method Specific Tables I (EPA 200.8), and II (EPA 6020). For information on statistical windows refer to *QA-SOP11896*.

4. Spike levels of batch QC are available in the LIMS and on T-MET-WI9082.

5. LOQs are available to analysts in the LIMS.

6. Check to make sure that all results are not> 90% of the Linear Range. If a sample reading is above 90% of the linear range, then reread the sample at a dilution sufficient to bring the sample concentration to approximately the middle of the calibration range.

7. Check that the **absolute** value of all nondetected analytes is less than the LOQ. A technical decision must be made as to whether a reread is warranted for readings <|-LOQ|.

8. Check for carryover between samples. Sample RSD >20%, with a concentration > the LOQ decreasing progressively over time (i.e., Reading 3<2<1). Flag any suspect samples for reread.

NOTE: Whenever a sample is encountered that exceeds the calibration range, the following sample must be checked for carryover for the over range analyte (s) and any suspect results

must be deselected and reanalyzed. During reanalysis the sample order must be modified as needed to avoid carryover from any over range sample(s).

9. For all EW (samples from public drinking water sources), check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL notify a verifier at once. An automated email is sent to the Client Service Representative and the state for the analytes listed below with the exception of Pb and Cu which follow the 90th percentile rule (the CSR tracks the lead and copper and notifies the supplier when necessary). Suppliers must be notified within 24 hours.

10. To avoid the risks associated with polyatomic interferences in wastewater and soil matrices all analytes must be analyzed on the collision cell ICP-MS instruments which include Agilent 7500, Equipment #11332, Agilent 7700x, Equipment #19204 and Agilent 7900, Equipment #27813).

a. Because EPA 200.8 rev. 5.4 does not currently allow the analysis of drinking water samples using collision cell ICP-MS all drinking water samples must be analyzed using a method with no gas mode for all analytes on either the Agilent 7500 or 7700. The following analytes are analyzed in drinking water samples: Be, Cr, Ni, Cu, As, Se, Ag, Cd, Sb, Tl, and Pb. Drinking water samples have also been historically free of the types of interferences that made alternate mass selection necessary; multiple masses are no longer used in the instrument method and only the masses referenced at the beginning of this procedure are used.

11. Check the internal standard level for the entire run. If the internal standard reading for any sample is out of the acceptance range of the S0 reading, evaluate whether this is a result of the sample matrix or drift from other samples in the run. Only reread the sample at a dilution if the internal standard appears to be out of range due to the sample matrix. For samples where an internal standard is above the acceptance range it is acceptable to reprocess the data using an alternate internal standard. This is the preferred approach in order to avoid raised reporting limits. The acceptance criteria differ based on the method being used. Refer to Table I, and II for the method acceptance criteria.

NOTE: The internal standard is added in equal concentration to all of the samples and standards via a dedicated line on the peristaltic pump. The analytical lines referenced to an internal standard report a corrected concentration value based on the ratio of analyte to internal standard intensities. All the calculations for determining concentration are based on Intensity Ratio (IR). The IR is defined as the background corrected intensity signal of the analyte line (Ia) divided by the internal standard value (Iis). IR= Ia/Iis.

12. For TCLP and SPLP samples, an MSA (method of standard additions) is required if:

a. The sample concentration falls between 80% and 100% of the regulatory limit.

b. If the TCLP or SPLP matrix spike (QA) recovers <20%, all samples in the leachate batch must be reanalyzed using the method of standard additions for that analyte.

13. When items 1 – 12 are complete, check the following:

a. All samples requiring reread/redigestion are listed on the reread/redigestion schedule forms.

b. The data are uploaded to LIMS via IDAT by the reviewer.

F. Verification process

1. Confirm that all required pieces of QC have been uploaded to LIMS and are within specification. If there is partial QC on the current run and the samples have been analyzed more than once, check to see if there are associated runs in the hold bin waiting for additional QC to be verified.

2. Choose method of verification. (Metals verification by run, verify by multiple elements per sample, or verify by individual element).

3. Check lab notes and project notes for each sample group.

4. If a technical decision or a client decision has been made to accept data that is not within specification, a nonconformance form *Q-EQA-FRM6869* must be filled out and signed by the investigator and a verifier. An electronic copy of the nonconformance form must be placed in the nonconformance forms folder located under the dept 22 folder on lldata/env. Quality Assurance reviews these forms on a monthly basis. Notify your supervisor each time that a nonconformance form has been generated.

5. Once all of the elements are verified for a digest, verify the digest number. Suite and tracking numbers will auto verify after all the metals analyses are verified.

Figure I - Daily Performance Acceptance Criteria

In Sensitivity	>300,000 cps

Mg Sensitivity	>40,000 cps
Pb Sensitivity	>100,000 cps
CeO/Ce	=0.03
Ba++/Ba+	=0.03
Background	<30 cps @ Mass 220
Net RSd	=%5.0

Table I - ICP-MS Metals by EPA 200.8 rev 5.4

*NOTE - PA EW SAMPLES ARE NOT ACCEPTED IF QC DOES NOT MEET CRITERIA. Redigest and reanalyze as needed if the acceptance criteria are not met.

Table I - ICP-MS Metals by EPA 200.8 rev 5.4			
QC	**Frequency**	**Acceptance**	**Corrective Action**
Tuning	Daily	No AMU diff. of >0.1 P.W. < 0.9 at 10% height (Agilent 7500 and 7700) %RSD <5 for masses used for tuning	Perform mass calibration for AMU. Adjust mass calibration for P.W.
Daily Performance	Daily	Evaluated for information only	Instrument maintenance and optimization as needed.
Single Point Calibration	Daily	The calibration must contain a blank and 1 standard	
Multi Point Calibration	Daily	$r^2 \geq 0.995$ Must contain a blank and 3 standards	Terminate run sequence and recalibrate. Data must not be reported for the analyte unless the correlation coefficient is =0.995
ICV	Immediately after calibration.	±10% of the true value If an element ICV is out high, accept element if the result < LOQ* .	Re-calibrate and re-analyze affected elements.
ICB		|ICB| < 3x IDL	

Table I - ICP-MS Metals by EPA 200.8 rev 5.4

	Immediately after the ICV.	If the ICB is out high, accept element results > 10x the ICB detection or < LOQ. If ICB is out low, only accept results > 10 x ICB detection*.	Re-calibrate and re-analyze affected elements.
LLC	Analyzed after ICB. Analyzed at the end of the run per client requirement.	± 50% of the true value. NA if samples are >10× the true value of the LLC. For LLC results >the high limit, samples <reporting limit can be accepted*	Data for that analyte must not be reported from the sample (reanalyze).
ICSA/ICSAB	ICSA: Analyzed after LLC ICSAB:Analyzed after the ICSA ICSA/ICSAB: Analyed at the end of the run per client requirement	± 20% of the true value for analytes that are spiked. |ICSA| or |ICSAB| must be <2× LOQ for analytes that are not spiked.	Data for that analyte must not be reported from the run (reanalyze).
CCV	Every 10 samples and at the end of the run.	±15% of the true value. If CCV is out, accept ND elements with |result| < LOQ*.	Data for that analyte must not be reported from the run (reanalyze).
Continuing Calibration Blank (CCB)	Every 10 samples and at the end of the run.	|CCB| must be <3x IDL If out high, accept element if the |result| < LOQ or > 10 x the detection in the CCB*. If out low, only accept results > 10 x the detection in the CCB*.	Data bracketed by the CCB for the affected analyte cannot be reported (reanalyze all samples in the bracketing blocks for that element).
PB	1 per analytical batch of 10 samples or less.	|PB| must be < 2.2x MDL. Does not apply to samples > 10x PB value or non-detects*.	Redigest all associated samples if the LCS is out of

Table I - ICP-MS Metals by EPA 200.8 rev 5.4

		EW samples are not accepted for any reason if the PB is out of specification.	specification low. If the LCS is out of specification high redigest samples that are greater than the LOQ. EW samples must be redigested.
LCS	1 per analytical batch of 10 samples or less. The LCS is spiked at or below the MCL for all primary drinking water metals. See QA-SOP11886 for MCLs.	Use statistical limits or the method limit of ±15%,, as indicated by the client requirement. If the LCS is out high and the sample result < LOQ the data can be taken*. EW samples are not accepted if the LCS is out of specification.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high redigest samples > LOQ. EW samples must be redigested.
LCSD	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±15%, as indicated by the client requirement. RPD < 20% If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken*.	Redigest all associated samples.
MS			

Table I - ICP-MS Metals by EPA 200.8 rev 5.4

	1 per analytical batch of 10 samples or less.	Use statistical limits or the method limit of ±30%, as indicated by the client requirement.	Data is flagged in the QC Summary and/or in the data package. If sample concentration <4X the spike added a PDS must be performed.
Duplicate (D)	1 per analytical batch of 10 samples or less.	If the samples are >5× the LOQ the RPD must be <20%	'*'-Flag the data package forms.
PDS	Must be prepared with each background sample. Evaluated when MS is out.	±15% of the background value.	Report % rec. on FormVB-IN.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations are >50 MDL.	The percent difference must be <10%	'E'- Flag data on DP Forms
Samples	Samples reading > 90% of the linear range must be reanalyzed. Elements reported as non-detect are accepted if the ICV/CCV is out of specification high and the sample is not < - LOQ.* RSD must be <20% for results >2× LOQ.		Sample is diluted and reanalyzed. EW must be re -analyzed Sample is reanalyzed.

Table I - ICP-MS Metals by EPA 200.8 rev 5.4			
Linear Range (LR)	Verify semi-annually.	±10% of the true value	
Internal Standards	Added to all samples by way of second pump channel.	Must be 60%-125% of the Calibration Blank Internal Standard	Reanalyze @ DF2

Table II - ICP-MS Metals by EPA 6020/6020A/6020B

Table II - ICP-MS Metals by EPA 6020/6020A/6020B					
QC	**Frequency**	**Criteria**			**Corrective Action**
		SW-846 6020	**SW-846 6020A**	**SW-846 6020B**	
Tuning	Daily	No AMU diff. of >0.1 P.W. < 0.9 at 10% height (Agilent 7500 and 7700) %RSD <5 for masses used for tuning			Perform mass calibrat Adjust mass calibratic
Daily Performance	Daily	Evaluated for information only			Instrument maintenar optimization as neede
Single Point Calibration	Daily	The calibration must contain a blank and 1 standard.			
Multi Point Calibration	Daily. Must contain a blank and 3 standards.	$r^2 \geq 0.995$.	$r^2 \geq 0.998$.	$r^2 \geq 0.995$.	Terminate run and re
ICV	Immediately after Calibration	%Recovery: 90-110%. If an element ICV is out high, accept element if the |result| < LOQ.			Re-calibrate and re-anal
ICB	Immediately after ICV	|ICB| <3× IDL	|ICB| <3× IDL	|ICB| 1/2 LOQ	Re-calibrate and re-ar elements: If the results are < 1 detection If the results were <l out low

Table II - ICP-MS Metals by EPA 6020/6020A/6020B

					Samples <LOQ can b is out high.
LLC	At the beginning of the run before ICSA/ICSAB. At the end of the run per client requirement.	%Recovery: 50-150% If out of range, accept element results >10× the true value of the LLC.	%Recovery: 70-130% If out of range, accept element results > CCV and CCV is in spec.	%Recovery: 80-120% If out of range, accept element results > CCV and CCV is in spec.	Data for that element reported from the run samples. If the LLC is out high <LOQ
ICSA	After LLC and at the end of the run per client requirement.	%Recovery: 80-120% |ICSA| < 2× LOQ for unspiked elements.	%Recovery: 80-120% |ICSA| < 2× LOQ for unspiked elements.	%Recovery: 80-120% |ICSA| < LOQ for unspiked elements.	Reanalyze samples that
ICSAB	After LLC and at the end of the run per client requirement.	%Recovery: 80-120% |ICSAB| < 2× LOQ for unspiked elements.	%Recovery: 80 -120% |ICSAB| < 2× LOQ for unspiked elements.	ICSAB is not required.	Reanalyze samples that
CCV	After every 10 samples and at the end of the run.	%Recovery: 90-110%. If out high, accept element if the |result| < LOQ.			Data bracketed by the samples with affected LOQ .
CCB	After every CCV, every 10 samples and at the end of the run	|CCB| <3× IDL	|CCB| <3× IDL	|CCB| < ½ LOQ	Data bracketed by the samples < 10x the CC Reanalyze if CCB was results were <LOQ Samples < LOQ can CCB is out high.
PB	1 per analytical batch of 20	|PB| must be < ½ LOQ. Not applicable if	|PB| must be < ½ LOQ. Not applicable if sample is > 10×	|PB| must be < ½ LOQ. Not applicable if	Sample may be re-an still out of specificatio associated samples.

Table II - ICP-MS Metals by EPA 6020/6020A/6020B

	samples or less.	sample is > 10× the PB reading or <LOQ.	the PB reading or <LOQ.	sample is > 10× the PB reading or <LOQ.	
LCS	1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, as indicated by the client requirement.			Sample may be re-an still out of specificatio associated samples. If the LCS is out high samples > LOQ.
LCSD	If required, 1 per analytical batch of 20 samples or less.	Must meet same criteria as LCS RPD < 20%			Same as LCS correcti Redigest samples if F specification.
MS/MSD	1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±25% as indicated by the client requirement. RPD must be <20%.			Data is flagged in the and/or in the data pa If sample concentrati added a PDS must be
D	1 per analytical batch of 20 samples or less.	If the samples are >5× the LOQ the RPD must be <20%.			Flagged in data packa Summary.
PDS	Must be prepared with each background sample. Evaluated when MS is not within specification.	±15% of the true value			The data is flagged in package.
Serial Dilution	Must be prepared with each background sample. Evaluated only when analyte concentrations	The percent difference must be <10%.			The data is flagged in package.

Table II - ICP-MS Metals by EPA 6020/6020A/6020B

	are >100X MDL.				
Samples		Sample reading must be within the linear range. RSD <20% for results > 2x LOQ. Elements <LOQ are accepted if the ICV/CCV is out of specification high, and the sample is not < - LOQ.			Sample is diluted and Sample is reanalyzed. Reanalyze for elemen meet this criteria.
Linear Range (LR)	Verified semi-annually.	±10% of the true value			Samples reading grea the calibration range reanalyzed.
Upper Linear Range	Analyzed once per run	NA	NA	±10% of the true value	Samples reading grea the calibration range reanalyzed.
Mid Range Check (1/2 the Upper Linear Range)	Analyzed once per run.	NA	NA	±10% of the true value	Reanalyze all element
Internal Standards	Added to everything through use of second pump channel	30%-120% for samples 80%-120% for ICV, CCVs, ICB, and CCBs. 30%-120% for LLC, ICSA, and ICSAB.	70-130% for samples. 80%-120% for ICV, CCVs, ICB, and CCBs. 30%-120% for LLC, ICSA, and ICSAB.	70-130% for samples. 80%-120% for ICV, CCVs, ICB, and CCBs. 30%-120% for LLC, ICSA, and ICSAB.	Reanalyze @ DF5. ICV/CCV/ICV/CCB: Te and reanalyze. LLC/ICSA/ICSAB: Rea

QA-SOP11880 Balance, Syringe, Pipette, and Labware Verification
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits
QA-SOP11901 Instrument Maintenance and Calibration
S-SS-WI10695 Liquid Sample Preservation, Sample Splitting, and Turbidity for metals by EPA Methods 200.7 and 200.8
T-MET-FRM8822 ICPMS Analysis
T-MET-WI11939 Digestion by EPA 200.8 for the Analysis of Total Recoverable Metals in Water by ICPMS
T-MET-WI12065 Working Instructions for Preparation of ICP-MS Solutions and Standards
T-MET-WI18026 Instrument Operations for Agilent 7500
T-MET-WI18027 Instrument Operations for Agilent 7700
T-MET-WI18028 Instrument Maintenance for Agilent 7500
T-MET-WI18029 Instrument Maintenance for Agilent 7700
T-MET-WI21589 Instrument Operations for Agilent 7900
T-MET-WI21590 Instrument Maintenance for Agilent 7900

End of document

Version history

Version	Approval	Revision information
7	29.JUN.2017	
8	11.SEP.2018	
9	21.MAR.2019	

E-15

Digestion of Aqueous Samples by SW846 Method 7470A, EPA 245.1
(Rev. 20; 03/21/2019; T-MET-WI11924)

eurofins	Always check on-line for validity.	Level:
Document number: **T-MET-WI11924**	**Digestion of Aqueous Samples by SW-846 Method 7470A, EPA 245.1**	**Work Instruction**
Old Reference: **1-P-QM-WI-9015082**		
Version: **20**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **21-MAR-2019**	Document users: **6_EUUSLA_Metals_Hg Prep, 6_EUUSLA_Metals_Hg Prep Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

LIMS ID
Analysis DOD - 5713, 5714

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Revision Log

Revision: 20	**Effective Date:**	This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Through out Document	Update	Updates SOP references to D4 numbers
Reference Modification	No change to chemistry	Removed section
Procedure A	Current Practice	Added procedure for samples that are not split. Added that hydroxylamine is added in the prep lab.

Revision:	19	**Effective Date:**	22.MAY.2017
Section	**Justification**		**Changes**
Revision Log			Removed revision logs up to the previous version

Revision:	19	**Effective Date:**	22.MAY.2017
	Formatting requirement per 1-P-QM-QMA-9017356		
Document Title	Correction of transcription error and clarification		Change EPA 254.1 to EPA 245.1 and add mercury analysis
Table of Contents	Unnecessary section		Removed section
Cross Reference	Higher level documents are not required to be referenced		Removed 1-P-QM-QMA-9015390
Purpose	Information contained within the scope		Removed section
Definitions	Optional section deemed unnecessary		Removed section
Personnel Training and Qualifications	Higher level documents are not required to be referenced		Removed 1-P-QM-QMA-9015390
Apparatus and Equipment	Enhancement		Moved LLENS into this section from the definitions
Procedure 6	Reflects current procedure		Added text pertaining to spiked leachate QA samples.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 7470A, September 1994

2. Method 245.1 (rev. 3), Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectroscopy, USEPA 600/R-94/111 May 1994.

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-MET-WI9084	Working Instructions for Preparation of Mercury Solutions and Standards

Scope

This procedure is used for automated and manual digestion of samples and standards for Mercury analysis in aqueous samples by SW-846 Method 7470A and EPA 245.1.

Basic Principles

The samples are digested with nitric acid, sulfuric acid, potassium permanganate, and potassium persulfate to oxidize mercury compounds to mercuric ions. Mercuric ions are reduced to mercury metal using stannous chloride. Mercury measurement is performed using the mercury cold vapor technique.

The DEENA Automated Sample Preparation System utilizes automated addition of reagents, heating, and filling to final volume to digest Mercury samples.

Interferences

Not applicable to this procedure.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

> **Nitric acid (HNO3)** – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes, or use the designated dispensing equipment.
>
> **Sulfuric acid (H2SO4)** – This acid is a strong oxidizing agent and can cause severe burns. Sulfuric acid spills are extremely slippery, adding to the danger. Always use in a fume hood, or use the desinated dispensing equipment. Never mix with concentrated HCl or concentrated KMNO4 to avoid a violent reaction (explosive splattering and extreme heat).
>
> **Hydrochloric acid (HCl)** – This acid can cause skin burns. Never mix HCI with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood, or use the designated dispensing equipment.
>
> When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X -A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each employee performing the digestion must work with an experienced employee for a period of time until they can independently perform digestions. Proficiency is measured through documented audits of the tasks listed as well as an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control samples (LCS's) that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Aqueous samples are collected in plastic or glass containers (drinking water sample volume is 1-L). Samples are preserved to a pH of <2 with nitric acid at collection and the pH is checked upon receipt and adjusted as necessary by Sample Support; samples that are pH adjusted at the laboratory must not be digested for a minimum of 24 hours. If samples fail to maintain a pH of <2 the Client Representative is notified for further direction. Samples are stored at 0 to 6°C, not frozen, prior to digestion. Samples must be analyzed within 28 days of collection.

Dissolved Mercury: Samples are to be filtered within 15 minutes of collection by the client. Clients may submit unpreserved bottles to the lab for dissolved mercury analysis. The sample is run through a 0.45 micron filter within 5 days of receipt and then preserved to a pH of <2 with HNO3.

Digested samples are stored in plastic bottles at room temperature. Store samples, standards, and digested samples separately.

Apparatus and Equipment

1. 50-mL polypropylene containers and covers (digestion vessels for block digestion) – certified clean and Class A equivalent

2. Environmental Express HotBlock (block digester) – adjustable and capable of maintaining a sample temperature of 95°C.

3. LLENS – the computer program that integrates a PC with an analytical balance to collect data directly from the balance. The program organizes the data and transmits the readings to the LIMS.

Reagents and Standards

1. For standards preparation, shelf life, and storage conditions, see ***T-MET-WI9084***. This form also has instructions for the initial preparation of Method Blanks, LCSs, Matrix Spikes and General Solutions.

2. Store all standards and reagents at room temperature. Label the container with the solution name, lot number, date prepared, the expiration date, the initials of the person preparing the solution, and the storage conditions.

3. Standard/ spiking concentration and reagent vendors are subject to change without notification.

4. Nitric acid, 70.0% to 71.0% HNO3, Fisher Trace Metal Grade reagent, 1.428 g/mL, or equivalent. Follow manufacturer's expiration date.

5. Sulfuric acid, 95.0% to 98.0%, H2SO4, 36 N, Fisher reagent, ACS, 1.84 g/mL, or equivalent. Follow manufacturer's expiration date.

6. Potassium permanganate, KMnO4, Baker Analyzed reagent, ACS, or equivalent. Follow manufacturer's expiration date.

7. Potassium persulfate, K2S2O8 Baker Analyzed reagent, ACS, or equivalent. Follow manufacturer's expiration date.

8. Sodium chloride, NaCl, J.T. Baker, Certified ACS, or equivalent. Follow manufacturer's expiration date.

9. Hydroxylamine hydrochloride, NH2OH·HCl, J.T. Baker, Certified ACS, or equivalent. Follow manufacturer's expiration date.

10. Stannous chloride, SnCl, Baker Analyzed reagent, ACS, or equivalent. Follow manufacturer's expiration date.

11. Hydrochloric acid, HCl, 36.5% to 38.0%, Fisher Trace Metal Grade reagent, 1.194 g/mL or equivalent. Follow manufacturer's expiration date.

Calibration

Not applicable to this procedure

Procedure

A. Manual hot block digestion

1. Turn block digester on and allow block to reach the Control Point setting that provides 95° ± 1°C sample temperature.

2. Before digestion, check the temperature with a calibrated thermometer and record in the logbook.

3. If the sample to be digested is not in split bin location:

   a. print label with the LLI Sample ID and batch number from LLENS and place on sample digestion containers.

   b. Shake sample well.

   c. Transfer 40 mL of well-mixed sample (or an aliquot diluted to 40 mL) into the polypropylene container. If a different final volume is necessary, adjust reagent volumes accordingly.

4. Prepare batch QC and calibration standards. See *T-MET-WI9084* for preparation instructions and concentrations. See the Quality Assurance/Quality Control section for the required batch QC.

NOTE: Exception - For leachate batches containing leachate spiked QA samples. Use one of the leachate spiked QA samples as the batch QC matrix spike and matrix spike dup, and proceed with the spiking procedures for the LCS according to ***T-MET-WI9084***.

6. Add 2 mL of H2SO4 and mix.

7. Add 1 mL of HNO3 and mix.

8. Add 6 mL of 5% KMnO4 solution and mix.

9. Allow the sample to stand for 15 minutes and then check sample for purple color.

   a. purple color does not persist for at least 15 minutes, perform a dilution on the sample beginning with a DF5.

   b. Continue diluting in increments of 5 or 10 until the purple color persists for at least 15 minutes.

10. Add 3.2 mL of 5% K2S2O8 solution and mix.

11. Place containers in block digester.

12. Place a calibrated thermometer in method blank container.

13. Put a polypropylene cover on each container. Remove after the samples reach 95° ± 1°C.

14. Heat for 2 hours in the block digester at 95° ± 1°C. The temperature is checked with a calibrated thermometer and recorded in the logbook.

15. Remove samples from block digester and cool.

16. Add 2.4 mL of 12% sodium chloride/hydroxylamine hydrochloride solution to reduce excess permanganate (color change is from purple to colorless). If the solution is not colorless, add reductant in 1-mL increments until KMnO4 is completely reduced.

17. Screw on cap and shake sample

18. Adjust the volume to 40 mL with reagent water and mix. Reserve for analysis.

NOTE: The block temperature is different than the temperature of the liquid being digested.

B. Block Digester Instructions:

1. Turn block digester on by pressing rocker switch located on the cord.

2. Wait about 8 seconds until controller display indicates current block temperature.

3. Press and hold STAR (*) key.

4. The display shows the Set Point Temperature.

5. The digits can be changed to the desired value by pressing the up and down arrow keys while holding the (*) key.

6. Confirm Control Point temperature is set to the block temperature that provides 95°C.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used for digestion. If necessary, adjust Control Point temperature to the proper setting.

NOTE: Polypropylene containers must not be heated above 130°C.

Calculations

Not applicable

Statistical Information/Method Performance

Not applicable

Quality Assurance/Quality Control

1. For 7470A, each digestion batch is up to 20 samples.

2. For 245.1, each digestion batch is up to 10 samples.

3. Each digestion batch must contain a method blank, LCS, U, D, and MS. If insufficient sample for a duplicate or per client requirement, an LCSD is added to the batch.

   a. Method Blank – equivalent to a Preparation Blank (PB).

   b. LCS/LCSD – Laboratory Control Sample/ Laboratory Control Sample Duplicate

   c. U – unspiked background sample

   d. D – duplicate

   e. MS (R) – Matrix Spike

   f. MSD (M) – Matrix spike duplicate

T-MET-WI9084 Working Instructions for Preparation of Mercury Solutions and Standards

End of document

Version history

Version	Approval	Revision information
19	13.OCT.2016	
19.1	22.MAY.2017	
20	19.MAR.2019	

E-16

Mercury in Aqueous, Solid and Tissue Samples by EPA 7471A, 7471B, 7470A, and 245.1 re/v 3 by Cold Vapor AA
(Rev. 17; 03/08/2019; T-MET-WI7965)

LIMS ID
Analysis DOD - 0259, 0159

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Tables

Revision Log

Revision	17	**Effective Date:**	This version
Section	**Justification**		**Changes**

Revision	17	**Effective Date:**	This version
Revision Log	Formatting requirement		Removed revision logs up to the previous version
Table I	Method Requirement		|PB| requirement to be < 2.2x MDL. ICB requirement to be < MDL.
Sample Analysis	Addition		Added filtration information as well as filtering the prep blank and laboratory control sample if a sample within the batch needs filtered.
Throughout	Clarification		Removed MCL from SOP. Reference the Compliance SOP *QA-SOP11886*
Procedure B.1.d.	Addition		Added information about removing cassettes.
Procedure B.2.b.	Update		Updated section to reflect use of Sequence Editor.
Sample Analysis A.1.a.	Removal		Removed preparation steps that are included in the Preparation procedure.
Sample Analysis A.3.b.	Addition		Added required steps for overnight shutdown to include cassettes.
QA/QC	Clarification/Enhancement		Clarified MSA requirements. Removed quarterly requirement for IDL studies.
Throughout	Clarification/Enhancement		Reorganized sections.

Revision:	16	**Effective Date:**	**27-NOV-2015**
Section	**Justification**		**Changes**
Revision Log	Formatting requirement per 1–P–QM-QMA-9017356		Removed revision logs up to the previous version
Personnel Training and Qualifications	Enhancement		Added information on IDOC and DOC options.
Table 1	Clarification for compliance		Added a note that the LCS is spiked at or below the MCL for drinking water.
Table 1	Clarification for compliance		Added new EW rule for the PB and LCS if they are out of specification data cannot be accepted for any reason.
Table 1 and 2	Clarification for compliance		PB requirements acceptance criteria updated.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 7470A, September 1994

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 7471B, February 2007.

3. Test Methods for Evaluating Solid Wastes, SW-846 Method 7471A, September 1994

4 Method 245.1 (rev. 3), Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectroscopy, USEPA 600/R-94/111 May 1994.

5. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-MET-WI11948	Preparation of Solids by EPA 7471A or B for Mercury Analysis
T-MET-WI11924	Digestion of Aqueous Samples by SW-846 Method 7470A, EPA 245.1
Q-EQA-FRM6869	Nonconformance Form
T-MET-WI9084	Working Instructions for Preparation of Mercury Solutions and Standards
QA-SOP11178	Demonstrations of Capability
QA-SOP11901	Instrument Maintenance and Calibration
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11886	Processing Regulatory Compliance (i.e. SDWA, NPDES) Samples

Scope

This method is used for determination of mercury in aqueous and solid samples. The optimum concentration range for this method is 0.2 to 5.0 ppb.

Matrices - EPA 7470A and EPA 245.1 are applicable to water analysis. EPA 7471A and EPA 7471B are applicable to soil and tissue analysis. EPA 245.1 is applicable to water analysis.

Limits of Quantitation (LOQs) are based on annual statistical evaluation of laboratory data and are subject to change. The current Method Detection Limits (MDLs) and LOQs are maintained in the LIMS, and are subject to change without notification.

Basic Principles

The Leeman Labs Mercury Analyzer utilizes continuous flow technology with drying of the sample vapor for the analysis of mercury by automated vapor generation. The reaction for the mercury analysis is a simple reduction reaction. The mercury is reduced with stannous chloride to liberate mercury metal and Tin (IV) chloride. An inert gas is used to sweep the volatile mercury into the absorption cell in the optical path of the atomic absorption spectrophotometer. The dry vapor enters one path of the optical cell, which has been optimized for fast response (small diameter), and sensitivity (long length).

Mercury is measured using a solid state detector with a wide dynamic range and a mercury source that delivers a stable source of emission at 254 nm. The signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions.

Definitions

1. Low Level Check Standard

2. MSA – Method of Standard Additions

3. SPLP - Synthetic Precipitation Leaching Procedure
4. TCLP – Toxicity Characteristic Leaching Procedure
5. QC Type Designators:
   a. U or US – unspiked background sample
   b. D – Sample Duplicate
   c. R – sample spike
   d. M – Sample spike duplicate
   e. PB/PBW/ PBS –Preparation Blank (Water/ Solid). Equivalent to a Method Blank (MB)

Interferences

Potassium permanganate is added to samples to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Take care to ensure that free chlorine is absent before the mercury is reduced and swept into the cell by using an excess of hydroxylamine sulfate (or chloride) reagent.

Copper has been reported to interfere; however, copper concentrations as high as 10 mg/kg had no effect on recovery of mercury from spiked samples.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Nitric acid (HNO3) – This acid can cause skin burns. Add nitric acid to samples in a hood to avoid exposure to toxic fumes.

Sulfuric acid (H2SO4) – This acid is a strong oxidizing agent and can cause severe burns. Sulfuric acid spills are extremely slippery, adding to the danger. Always use in a fume hood. Never mix with concentrated HCl or concentrated KMNO4 to avoid a violent reaction (explosive splattering and extreme heat).

Hydrochloric acid (HCl) – This acid can cause skin burns. Never mix HCI with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds. Samples that contain dust may be hazardous. Open in a fume hood.

Samples that may contain cyanide require special precautions to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X -A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the system to set up sequences, perform the calculations, interpret raw data, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample. Refer to *QA-SOP11178* for more guidance on these options.

Sample Collection, Preservation, and Handling

Aqueous samples are collected in plastic or glass containers, preserved to a pH of <2 with nitric acid. Samples must be analyzed within 28 days of collection.
NOTE: Regulatory Drinking Water samples (EW) are collected in 1-L plastic or glass containers and must be analyzed by EPA 245.1. Drinking water samples are not analyzed for dissolved mercury.

Solid samples are collected in glass containers and stored at 0° to 6°C not frozen. Samples must be analyzed within 28 days of collection.

Dissolved Mercury: Samples to be analyzed for soluble mercury requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45 micron filter within 5 days of receipt and then for aqueous samples**,** samples are collected in plastic containers and preserved to a pH of <2 with HNO3.

Store sample digestates in plastic bottles at room temperature. Store standards and digestates separately.

Apparatus and Equipment

1. Hydra II Mercury Analyzer

2. Leeman Labs Envoy software – a windows based program to help navigate the software.

Reagents and Standards

A. Store all standards and reagents in polyethylene or glass containers at room temperature. Label the container with the solution name, lot number, date prepared, the expiration date, the initials of the person preparing the solution, and the storage conditions.

NOTE: Standard/ spiking concentration and reagent vendors are subject to change without notification.

B. Reagents use the following or equivalent:

1. Nitric acid, 70.0% to 71.0% HNO3, Fisher Trace Metal Grade reagent, 1.428 g/mL; Store in glass container at room temperature. Follow manufacturer's expiration date.

2. Sodium chloride, NaCl, J.T. Baker, Certified ACS. Store in plastic container at room temperature. Follow manufacturer's expiration date.

3. Hydroxylamine hydrochloride, NH2OH·HCl, J.T. Baker, Certified ACS. Store in plastic container at room temperature. Follow manufacturer's expiration date.

4. Reagent Water

5. Stannous chloride solution, 10% SnCl, Baker Analyzed reagent, ACS. Store in plastic container at room temperature. Follow manufacturer's expiration date.

6. Hydrochloric acid, HCl, 36.5% to 38.0%, Fisher Trace Metal Grade reagent, 1.194 g/mL or equivalent. Store in glass container at room temperature. Follow manufacturer's expiration date.

C. For the preparation of calibration blanks, ICBs, CCBs, calibration standards, ICVs, CCVs, CRAs, Method Blanks, LCSs and Matrix Spikes solutions, see Form *T-MET-WI9084*.

D. General solutions - See Form *T-MET-WI9084*.

Calibration

Leeman Labs Hydra II Mercury Analyzer

1. The software program has been developed to check the correlation coefficient of the curve, run appropriate ICV and CCVs at proper intervals, and check the percent recoveries of the ICV and CCVs.

2. Due to the instrument software limitations, the calibration blank is included in the correlation coefficient calculation.

3. A recalibration and reread of any associated samples is required for any checks that fall outside the windows.

4. For the frequency, acceptance criteria and corrective action see Table I and Table II.

5. For the preparation of calibration standards, see *T-MET-WI9084*.

6. The calibration is part of each run from Sequence Editor from the LIMS.

Procedure

A. Sample Digestion

1. Aqueous samples are digested according to SOP *T-MET-WI11924*.

2. Solid samples are digested according to SOP *T-MET-WI11948*.

B. Leeman Labs Hydra II Mercury Analyzer Operation

1. Instrument Setup

a. Turn ON the power to the instrument (switch in the back) and computer.

b. Ensure that Argon supply is set to 15 psi.
c. Double click the Envoy icon on the desktop to initialize the instrument software.

d. Put on the peristaltic pump cassettes. Do not over tighten. At the end of shift, remove them from the pump.

e. Place levers in the 1 o-clock position to avoid stalling the pump.

f. Check that the rinse bottle is full and Luer connections are tight. Only a 'light' finger tightening is required. Refill the rinse tank with a 2.0% Hydrochloric Acid (HCl) solution. For preparation of 2.0% HCl solution, see *T-MET-WI9084*.

g. Check that the 10% stannous chloride bottle is full and Luer connections are tight. Only a 'light' finger tightening is required. For preparation of 10% stannous chloride, see *T-MET-WI9084*.

h. Click the start icon on the Tool Bar to turn on the peristaltic pump and set the gas flow to method programmed conditions.

i. Check to see that the lamp, pump and gas turn on. If necessary, open the Method/Instrument Control Panel and turn them on and set appropriate parameters.

j. When the pump is turning, tighten the cassettes by lowering the levers to a horizontal position. Allow 10 minutes for lamp and pump equilibration.

k. Inspect all system connections for leaks.

l. The system is now ready to be optimized for automated analysis.

2. Autosampler and Run Setup

a. Create the run sequence using Sequence Editor found in the IDAT menu in eLIMS.

b. Click on the Sequence Menu, then Open, and then choose your sequence file created from Sequence Editor

c. Each row represents a cup location on one of the racks and its graphical representation updates in the lower "Rack" graphic whenever the Update button is clicked

d. Edit the sequence, if needed. The samples, standards, and Quality Control samples are automatically populated by Sequence Editor.

e. Click the "Update" button to when all samples are entered to populate the navigation tree to the left with the proposed run sequence.

Sample Analysis

1. Filtering Samples

a. Samples that are cloudy or have particulate suspended in solution must be filtered prior to analysis.

b. Using the Filter Mate filtration device, filter the samples and if the filtrate remains cloudy, filter again. Difficult matrix samples or samples with limited volume must be filtered using a 10-mL sterile disposable syringe fitted with a 0.45 um PTFE syringe filter.

c. If any samples are filtered, the PB and the LCS on the batch must be filtered.

d. Document all filtrations on the run cover sheet.

2. Dilutions

a. Dilute samples with appropriate matrix matching solution when necessary to yield a response that falls within the calibration range.

b. Report the results for the least dilute sample where the concentration measured is within the acceptable calibration range.

3. Analysis of samples with Leeman Labs Hydra II Mercury Analyzer

a. Click the "Run Sequence" icon to start the run. If a dialog appears after the Run Sequence icon is clicked, follow the instructions of those prompts to resolve issues before running the sequence.

b. The system adds Stannous chloride to the samples via a "Y" connection in the pump tubing. The peristaltic pump then carries the sample/stannous mix to the liquid gas separator. Argon gas is bubbled through the liquid and used to transport the volatile mercury into the detector. The mercury is reduced with stannous chloride to liberate mercury metal and Tin (IV) chloride.

NOTE: Detailed instructions for the complete instrument setup are found in the Leeman Labs Hydra II Automated Mercury Analyzer Manual.

4. Overnight Shutdown

a. Click on the "sleep" icon to stop argon flow and pump

b. In sleep mode the pump is cycled on periodically to relieve pressure points where the rollers contact the tubing.

c. Remove peristaltic pump cassettes

Never leave bottle of reductant and rinse connected to the instrument if the pump clamps are released because siphoning can occur and cause damage to the instrument.

5. Long-term shutdown (more than 3 days of no operation).

a. Place reductant tubing and rinse tubing into a beaker of reagent water.

b. Run pump until system is flushed of reagents. Send autosampler tip to air.

c. Remove reductant and rinse tubing from beaker to allow the aspirating of air.

d. Run pump until system is flushed of liquid. Some liquid does remain in the liquid/gas separator.

e. Turn OFF the pump.

f. Close Envoy program and power down the instrument.

g. Shut OFF the power to the computer and monitor.

h. Shut down argon gas flow.

Calculations

1. Final Result

   a. Water sample

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

   b. Solid sample (mg/kg)

$$\text{Instrument Reading} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

2. Relative percent different (RPD)

$$RPD = \frac{S - D}{(S + D)/2} \times 100$$

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

$$\%\,Recovery = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{\left(\sum X^2 - \frac{(\sum X)^2}{N}\right)\left(\sum Y^2 - \frac{(\sum Y)^2}{N}\right)}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\% \, Difference = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Methods of standard additions (MSA)

Take 4 identical aliquots of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution. Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y- intercepts. The x- intercept is the result.

$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i)/n}{\sum x_i^2 - (\sum x_i)^2/n}$$

$$\text{Y-Intercept} = b = \bar{y} - m\bar{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum\{(x_i - \bar{x})(y_i - \bar{y})\}}{\sqrt{\left[\sum (x_i - \bar{x})^2\right]\left[\sum (y_i - \bar{y})^2\right]}}$$

The correlation coefficient (r) for the least squares fit must be ³0.995. If the r value is <0.995, the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is <0.990, the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

Statistical Information/Method Performance

Generate MDLs and LOQs according to *QA-SOP11892*. The department supervisor maintains annual study data.

QC limits for MS/MSD, and LCS/LCSD are established through statistical analysis of historical data. The limits are maintained in the LIMS for the relevant analysis numbers.

The department supervisor requests that a Quality Assurance Specialist update to the LIMS as needed. Update the department database via a download from the LIMS.

Quality Assurance/Quality Control

Batch Quality Control

1. For the preparation and concentrations of Batch Quality Control see *T-MET-WI9084*.

2. For the frequency, acceptance criteria and corrective action see tables III and IV.

3. For EPA 7470A, 7471A, and 7471B, a digestion batch is up to 20 samples. Each batch must contain a PB, LCS, U, D, MS and MSD (an LCSD is used if there is insufficient sample for an MSD).

4. For EPA 245.1 Rev 3, a digestion batch is up to 10 samples. Each batch must contain a PB, LCS, US, D, and MS (an LCSD is used if there is insufficient sample for an MSD).

Data Review

A. Raw Data Quality Checks

1. Confirm that the batch and cover sheets are correctly labeled, dated, and signed where necessary.

2. Review the batch sheet, project notes and lab notes with the incomplete list for special comments and due dates.

3. Check that the run protocol has been selected correctly.

4. Refer to the calculation section of this SOP for calculations used for Hg analysis.

5. Refer to Tables I and II for instrument calibration and verification frequency, acceptance criteria and corrective action.

6. Refer to Tables III and IV for batch QC frequency, acceptance criteria and corrective action. For information on statistical limits see *QA-SOP11896*.

7. Each analytical run must have a QC review attached. All samples on the run must be listed on the QC review with notation as to whether the sample was verified or needed to be redigested/reanalyzed. The verifier must document on the QC review if any sample(s) were selected/deselected.

8. For spike levels of run and batch QC, see *T-MET-WI9084*.

9. LOQs are available to analysts in the LIMS.

10. Check to make sure that all results are within the calibrations range. If a sample reading is above the calibration range, then reread the sample at an appropriate dilution.

11. Check that the negative value of NDs are greater than negative the LOQ. A technical decision must be made as to whether a reread is warranted for readings ≤ -LOQ.

12. For TCLP and SPLP samples, an MSA (method of standard additions) is required if:

a. The sample concentration falls between 80% to 100% of the regulatory limits AND the MS Recovery is <50%.

13. For all EW samples (samples from public drinking water sources); check the results against the MCL (maximum contaminant level). If an analyte exceeds the MCL, notify a verifier at once so that the supplier can be notified. The verifier must contact the Client Service Representative, who must then notify the Supplier. Suppliers must be notified within 24 hours. Reference *QA-SOP11886*.

B. When raw data checks are complete, check the following:

1. All samples requiring redigestion are listed on the redigestion schedule.

2. Redigest request forms are clipped to the front of the run.

3. The data are uploaded to eLIMS-EP via IDAT by reviewer then verified from eLIMS-EP by a verifier.

4. The data packet is placed in the verification bin.

C. Verification process

1. Confirm that all required pieces of QC have been uploaded to eLIMS-EP and are within specification.

2. If there is partial QC on the current run and the samples have been analyzed more than once, check to see if there are associated runs in the hold bin waiting on additional QC to be verified.

3. In eLIMS-EP, choose method of verification. (Metals verification by run or verify by individual element).

4. Ensure that all lab notes and project notes were followed.

5. Non-compliant data can be reported only after all required corrective actions have been taken. Document the nonconformance using *Q-EQA-FRM6869*.

6. Once all of the elements are verified for a digest, verify the digest number. Associated tracking numbers or suite numbers will be auto-verified after all of the metals are verified.

Instrument Maintenance and Repair

A. Routine Maintenance

1. Replace the pump tubing as needed under normal daily usage.

2. On an as needed basis, check the optical cell and windows, and if needed, clean the optical cell.

   a. Wipe the optical cell with a soapy solution (one drop of liquid Ivory soap to 500 mL reagent water) and warm tap water.

   b. Rinse with reagent water and dry. To speed the drying of the optical cell, connect the heater plug to the optical cell with the windows off for several minutes.

   c. Clean the quartz windows with methanol and a piece of lens paper.

3. Document any maintenance in the Mercury maintenance logbook located next to the instrument.

NOTE: Detailed instructions for the maintenance and troubleshooting of the Leeman Labs Mercury Analyzer can be found in the Leeman Labs Hydra II Mercury Analyzer Manual.

B. Taking an instrument/analysis out of service/returning an instrument/analysis to service.

NOTE: In the event of an equipment failure, perform the steps in SOP *QA-SOP11901*:

1. Document the nature of the failure in the maintenance logbook.

2. Document how and when the defect was discovered.

3. Notify a supervisor or experienced analyst to determine a person who can decide on appropriate action to take.

4. The instrument must be clearly tagged as Out of Service. The tag must contain the following information:

   a. Date taken out of service.

b. Employee who took the instrument out of service.

c. Reason for tagout.

5. The date taken out of service and the date returned to service must be documented in the logbook.

6. Document any corrective action that was taken and the result of that corrective action (i.e., system calibration within specifications, etc.) to bring the equipment back into service.

7. Supervisory personnel must perform a documented evaluation and review of instrumentation/equipment where a major or uncommon failure has occurred to assess the potential impact the failure could have on the calibration and/or qualification of the instrument.

8. After a repair, document whether the function has been fixed. Calibration or verification activities are to be performed before the instrumentation is put back into service.

Tables

Table I: Calibration Requirements and Corrective Actions EPA 245.1 Rev 3

	Frequency	**Acceptance**	**Corrective Action**
Calibration	The calibration contains a blank and 5 standards.	Correlation coefficient >0.995	If the correlation coefficient is not met, confirm instrument conditions (i.e. check pump tubing and gas liquid separator). Reanalyze the curve, if the correlation coefficient is acceptable, proceed with sample analysis. If the correlation coefficient is not met after reanalysis, redigest and reanalyze the curve and all associated samples.
Initial Calibration Verification (ICV)	Must be analyzed immediately following the calibration.	±5% of True Value If the ICV is out of specification high report the elements that are < LOQ.	For samples >LOQ, the instrument must be recalibrated and samples reanalyzed. Confirm instrument conditions (i.e. check pump tubing and gas liquid separator). Reanalyze the

		For elements > LOQ, data cannot be reported. EW Samples are not acceptable if the ICV is out of spec.	ICV, if the recovery is acceptable, proceed with sample analysis. If the acceptance criteria are not met after reanalysis, redigest and reanalyze the curve and all associated samples. EW samples must be reported with an acceptable ICV.
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV	Must be <MDL	Data cannot be reported from the run (reanalyze).
Contract Required Detection Limit (CRA)	Must be analyzed immediately after the ICB	±50% of the true value	Data cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed at the frequencyof every 10 samples and at the end of the analytical run.	±10% of the true value If the CCV is out of specification high and the sample is not < - LOQ, accept samples that report as non-detect. EW samples must be reported with acceptable bracketing CCV.	Data bracketing the CCV cannot be reported from other samples on the run (reanalyze)*.
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCVs at a frequency of every 10 samples.	Must be <MDL	Data bracketing the CCB cannot be reported from the run (reanalyze)*

Table II: Calibration Requirements and Corrective Actions EPA 7470A, 7471A/B			
	Frequency	**Acceptance**	**Corrective Action**

Calibration	The calibration contains a blank and 5 standards.	Correlation coefficient >0.995	If the correlation coefficient is not met, confirm instrument conditions (i.e. check pump tubing and gas liquid separator). Reanalyze the curve, if the correlation coefficient is acceptable, proceed with sample analysis. If the correlation coefficient is not met after reanalysis, redigest and reanalyze the curve and all associated samples.
Initial Calibration Verification (ICV)	Must be analyzed immediately following the calibration.	±10% of True Value If the ICV is out of specification high, report the elements that are < LOQ (elements > LOQ require corrective action)	Confirm instrument conditions (i.e. check pump tubing and gas liquid separator). Reanalyze the ICV, if the recovery is acceptable, proceed with sample analysis. If the acceptance criteria are not met after reanalysis, redigest and reanalyze the curve and all associated samples. The instrument is recalibrated and EW samples reanalyzed.
Initial Calibration Blank (ICB)	Must be analyzed immediately following the ICV	Must be <lLOQl	Data cannot be reported from the run (reanalyze).
Contract Required Detection Limit (CRA) Limit of Quantitation Check Standard	Must be analyzed immediately after the ICB	For 7470A and 7471A: ±50% of the true value For 7471B: ±30% of the True Value	Data cannot be reported from the run (reanalyze).
Continuing Calibration Verification (CCV)	Must be analyzed after every 10 samples and at the end of the analytical run.	±20% of the true value If the CCV is out of specification high, samples that are <	Data bracketing the CCV cannot be reported from other samples on the run (reanalyze)* Samples >LOQ are reanalyzed.

		LOQ may be reported. EW Samples are not accepted if out of spec.	EW samples are not accepted if the CCV is out of specification.
Continuing Calibration Blank (CCB)	Must be analyzed immediately following CCVs at a frequency of every 10 samples.	Must be <ILOQI	Data bracketing the CCB cannot be reported from the run (reanalyze)*

* If the CCV/CCB are out of specification, it can immediately be re-read in duplicate. If both CCV/CCBs are within specification, the data from the last acceptable verification is reanalyzed. If one or both CCV/CCBs are still out of specification, then the run is terminated and the samples after the last good CCV/CCB must be reanalyzed on a new run.

Table III: Batch QC Criteria and Corrective Actions for EPA 245.1 Rev 3			
	Frequency	**Acceptance**	**Corrective Action**
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	IPBI must be < 2.2x MDL. Not applicable if analyte reading in the sample is >10× the PB reading or non-detect. EW samples are not accepted for any reason if the PB is out of specification.	Redigest all associated samples. EW samples must be redigested.
Laboratory Control Standard (LCS)	Must be prepped at a frequency if 1 per analytical batch of 10 samples or less. *Note: The LCS is spiked at or below the MCL for drinking water. See *QA-SOP11886* for MCL level.	Use statistical limits, or the method limit of ±15%, as indicated by the client requirement. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken. EW samples are not accepted if the LCS is out of specification.	Redigest all associated samples if the LCS is out of specification low. If the LCS is out of specification high, redigest samples that are greater than the LOQ. EW samples must be redigested.
Laboratory Control Standard	If insufficient sample volume is submitted to perform batch QC	Use statistical limits, or the method limit of ±15% As indicated by the client	Redigest all associated samples if the LCSD is out of specification low.

Duplicate (LCSD)	then a LCSD is prepped at a frequency of 1 per analytical batch of 10 samples or less. *Note: The LCS is spiked at or below the MCL for drinking water. See *QA-SOP11886*. for MCL level.	requirement. If the LCSD is out of specification high and the sample result is less than the LOQ the data can be taken. EW samples are not accepted if the LCS is out of specification. RPD must be <20%.	If the LCSD is out of specification high, redigest samples that are greater than the LOQ. EW samples must be redigested. Redigest if RPD is out of specification
Matrix Spike (MS)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	Use statistical limits or the method limit of ±30% whichever is tighter. (PW,EW) Use statistical limits or the method limit of ±20% whichever is tighter (WW)	The data is flagged in the QC Summary and/or in the data package.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 10 samples or less.	If the samples are >5× the LOQ the RPD must be <20%. If either the sample or duplicate is <5× the LOQ the difference between the two values must be <LOQ.	The data is flagged in the QC Summary and/or in the data package.

Table IV: Batch QC Criteria and Corrective Actions for EPA 7470A, 7471A and 7471B

	Frequency	**Acceptance**	**Corrective Action**
Preparation Blank (PB)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	IPBI must be < ½ LOQ . For 7470A and 7471A: Not applicable if analyte reading in the sample is >20× the PB reading or <LOQ. For 7470B and 7471B: Not applicable if analyte reading in the sample is >10× the PB reading or <LOQ.	Redigest all associated samples.
Laboratory Control	Must be prepped at a frequency if 1 per	Use statistical limits or the method limit of ±20%, as	Redigest all associated samples if the LCS is out of specification low.

Standard (LCS)	analytical batch of 20 samples or less.	indicated by the client requirement. If the LCS is out of specification high and the sample result is less than the LOQ the data can be taken.	If the LCS is out of specification high, redigest samples that are greater than the LOQ.
Laboratory Control Standard Duplicate (LCSD)	If insufficient sample volume is submitted to perform batch QC then a LCSD is prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20%, as indicated by the client requirement. If the LCSD is out of specification high and the sample result is less than the LOQ the data can be taken. RPD must be <20%.	Redigest all associated samples if the LCSD is out of specification low. If the LCSD is out of specification high, redigest samples that are greater than the LOQ. Redigest if RPD is out of specification
Matrix Spike (MS)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	Use statistical limits or the method limit of ±20% whichever is tighter.	The data is flagged in the QC Summary and/or in the data package.
Duplicate (D)	Must be prepped at a frequency of 1 per analytical batch of 20 samples or less.	If the samples are >5× the LOQ the RPD must be <20%. If either the sample or duplicate is <5× the LOQ the difference between the two values must be <LOQ.	The data is flagged in the QC Summary and/or in the data package.

Q-EQA-FRM6869 Nonconformance Form
QA-SOP11178 Demonstrations of Capability
QA-SOP11886 Processing Regulatory Compliance (i.e. SDWA, NPDES) Samples
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits
QA-SOP11901 Instrument Maintenance and Calibration
T-MET-WI11924 Digestion of Aqueous Samples by SW-846 Method 7470A, EPA 245.1
T-MET-WI11948 Preparation of Solids by EPA 7471A or B for Mercury Analysis
T-MET-WI9084 Working Instructions for Preparation of Mercury Solutions and Standards

End of document

Version history

Version	Approval	Revision information
16	27.NOV.2015	
17	04.MAR.2019	

E-17

Separatory Funnel Extraction Procedure for HRMS Analysis in an Aqueous Matrix Using Method 1613B, 8290A, 1668A, and 1668C (Rev. 4; 12/05/2018; T-HRMS-WI12032)

eurofins	Always check on-line for validity.	Level:
Document number: **T-HRMS-WI12032**	**Separatory Funnel Extraction Procedure for HRMS Analysis in an Aqueous Matrix Using Method 1613B, 8290A, 1668A, and 1668C**	**Work Instruction**
Old Reference: **1-P-QM-WI-9038109**		
Version: **4**		Organisation level: **5-Sub-BU**
Approved by: **UKL3** Effective Date **05-DEC-2018**	Document users: **5_EUUSLA_HRMS_Manager, 6_EUUSLA_HRMS_Analyst, 6_EUUSLA_HRMS_Data_Reviewers, 6_EUUSLA_HRMS_Management_Team, 6_EUUSLA_HRMS_Sample_Prep**	Responsible: **5_EUUSLA_Specialty Services_Manager**

LIMS ID
Analysis 10914, 13235, 14069

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Revision Log
Reference
Cross Reference
Scope
Basic Principles
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Preparation of Glassware
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

Revision 4		**Effective Date:**	This version
Section	**Justification**		**Changes**
Revision Log	Formatting requirement		Removed revision logs up to the previous version
Procedure Section 6 step j	Clarity		PCDD/PCDF changed to PCB to accurately reflect what analysis is performed on the extract.
Reagents and Standards	Addition of a standard		Added cleanup standard spiking solution
Procedure Section B.1	Consistency		Cleanup spiking solution was changed to cleanup standard spiking solution for consistency

Revision: 3	**Effective Date:**	This version
Section	**Justification**	**Changes**
Apparatus and Equipment	Reflects current practice	Changed balance capability to 0.01 gram. Removed water bath, sonic bath, and glass etching pen. Added GC vials, GC caps and Chromacol vial caps.
Procedure	Reflects current practice	Updated procedure for cleaning sodium sulfate. Updated procedure for preparation of acid silica gel, basic silica gel.
Procedure B.2.a	Reflects current practice	Added section on use of automated sample weighing program.
Procedure B.2.c	Reflects current practice	Updated spike amounts to reflect dioxin/furan and PCB congeners.
Procedure B.3.c	Reflects current practice	Added use of a mini-separatory funnel or centrifugation to break up emulsions.
Procedure C.1	Reflects current practice	Changed order of column prep. Alumna column first, followed by multi-layer silica gel column
Procedure C.2.c	Reflects current practice	Updated amount of acid silica gel to 1 tablespoon
Procedure C.5	Reflects current practice	Updated cleanup section for dioxins and furans
Procedure C.6	Reflects current practice	Updated cleanup section for PCB congeners

Reference

1. EPA Method 1613B, Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, October 1994.

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 8290A, Polychlorinated Dibenzo-p -Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS), February 2007 Rev 1.

3. EPA Method 1668C, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, April 2010

4. EPA Method 1668 Revision A, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, August 2007

5. USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures, Quality Assurance, Fifth Edition

6. *Chemical Hygiene Plan,* current version.

Cross Reference

Document	Document Title

Document	Document Title
Analysis #10915, 11031, 11645, 11650, 12935, 12936, 12937, 13232, 13233	Determination of Tetra- Through Octa-Chlorinated Dioxins and Furans using HRGC/HRMS by EPA 1613B or SW-846 8290A
Analysis #12154, 12429, 13707, 13708	PCB Congeners by Method 1168 HRGC/HRMS in Aqueous and Solid Matrices
Analysis #11030,13234	Soxhlet Extraction Procedure for HRMS Analysis in a Solid Matrix
T-HRMS-WI9446	Standards Management in the High Resolution Mass Spectrometry Laboratory
T-HRMS-WI9485	Glassware Cleaning for HRMS Extractions

Scope

This method is suitable for the extraction of dioxins/ furans and PCBs, at ppt (parts per trillion) levels in water, wastewater, and water soluble matrices that are not prone to emulsions. The detection limits and quantitation levels in this method can be dependent on the level of interferences observed in the sample analysis.

Basic Principles

A 1-L portion of sample to be analyzed is placed into a 2-L separatory funnel. Isotopically labeled standards are added to each sample to monitor recovery. The sample is extracted with methylene chloride. The extract is dried, concentrated, and if necessary, cleaned using various column cleanups.

Interferences

Method interferences may be caused by interfering compounds in the sample matrix, impurities in solvents, reagents, glassware, or other hardware used in sample processing. All glassware is rinsed with solvent before use and a method blank is performed with each batch of samples to demonstrate that the extraction system is in control.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

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. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves.

Since the extracts are concentrated on a steam bath, caution must be exercised while working around this apparatus.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. Additional precautions to those outlined in the *Chemical Hygiene Plan* are required in the laboratory used to perform the extraction analysis. Waste streams have been defined to handle dioxin and PCB sample processing waste:

A. Flammable Dioxin Extracts – This waste stream consists of sample extracts in GC vials.

B. Dioxin Samples Waste – This waste stream consists of empty used ampoules, disposable glass pipettes, gloves, spent column adsorbents, post extraction solid sample residue, and other incidental consumable items used in the processing of dioxin/furan samples.

C. Flammable GC Vials w/ PCBs- This waste stream consists of sample extracts in GC vials

D. Samples with PCBs – This waste stream consists samples with PCBs greater than 50 ppm.

Solvents that are used as a secondary cleaning rinse for cleaned glassware are disposed of according to solvent disposal procedures outlined in the *Chemical Hygiene Plan*.
Expired dioxin/furan and PCB standards are routinely collected for disposal through a lab pack process and arrangements with the lab waste disposal vendor.

Lab coats are designated for dedicated use in the dioxin/furan sample preparation lab. These lab coats are not worn for work in other labs, and lab coats worn for work in other labs are not worn in the dioxin/furan sample prep lab.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Initially, each technician performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC)

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples are collected in 1 liter amber glass containers. Aqueous samples are to be tested for residual chlorine and pH upon sample receipt. If residual chlorine is present, add 80 mg of sodium thiosulfate ($Na_2S_2O_3$) per liter of water. If the pH is greater than 9, adjust to pH 7 to 9 with sulfuric acid. This procedure is performed by the Sample Support Department. All samples are stored refrigerated at 0° to 6°C, not frozen, in the dark prior to extraction. Samples are to be extracted within 30 days of sample collection and analyzed within 45 days of extraction for dioxin/furans, and extracted within 30 days of sample collection and analyzed within 40 days of extraction for PCBs. All drinking water sample extracts must be analyzed within 40 days of extraction. Unused portions of samples and sample extracts may be stored in the above manner for up to six months after sample receipt to allow further analysis, if necessary.

Analysis 14069 requires a 2L sample volume be extracted. Attachment I details the requirements.

Apparatus and Equipment

1. 2-L separatory funnel for extracting organic components from an aqueous matrix

2. Kuderna-Danish (K-D) assembly with appropriate ampule for concentrating the solvent used during the extraction

3. Water bath – VWR/LLI Model #1127 or equivalent

4. Graduated cylinders – Class A, assorted sizes

5. Pipettes – Class A, assorted sizes

6. Pipettes – Disposable

7. Balance – Capable of weighing to 0.01 g

8. Automatic shaker – Capable of holding 2-L separatory funnels

9. Centrifuge – Thermo Scientific Sorvall Legend T3 Centrifuge or equivalent

10. Sodium sulfate columns with SS screens, no frits

11. Wash bottles – Teflon

12. Teflon boiling chips, Fisher Scientific, 09-191-20, or equivalent, soxhlet extracted in toluene for minimum of 4 hours.

13. Custom 600 mL Buchner funnel with 3 inch SS screen, QFN-600-SP, Quark Glass

14. Glass fiber filter paper, 90 mm, type GF/C or 934-AH

15. 2-L filter flask

16. Centrifuge – Fisher Scientific FS110 (or equivalent)

17. Column adsorbent preparation tube, Quark Enterprises, QD-100-4550-ROD, Top Rodaviss Jt., 45/50., or equivalent

18. Preparation tube adapter, Quark Enterprises, QA-45-ROD, Adapter #7 Top Internal Thread with Bushing, 45/50 Male Rodaviss Joint, or equivalent

19. 2000 mL solvent reservoir, Quark Enterprises, QCH-8-7, or equivalent

20. 4 liter filter flask, Quark Enterprises, QF-7-4L-4550, or equivalent

21. Tube Furnace with controller, Lindberg Blue M Hinged Tube Furnace, Model HTF55342C, or equivalent

22. Bottle roller mixer, Wheaton, W348924-A, or equivalent

23. Glass bottle, 1 gallon, 1qt , wide mouth

24. Desiccator cabinet

25. 300 mm X 0.625" OD Column, Quark Enterprises, QD-300, with 24/40 outer one end and 14/20 inner other end w/ss screen

26. Cleanup Column #1, Quark Enterprises, QSGC-1-32, Version 3

27. 240 mm X 0.625" OD Column, Quark Enterprises, QD-240, with 14/20 outer at one end and 14/20 inner other end with ss/screen

28. Cleanup Column #2, Quark Enterprises, QSGC-2-32, Version 4

29. Teflon/Silicone seal ring, Quark Enterprises, QSR-32-16

30. Red Cap with hole, Quark Enterprises, QCP-32-RMD

31. Large acid silica gel column, Quark Enterprises, QD-400-10.

32. 250 mL solvent reservoir with 24/40 joint, Quark Enterprises, QCH-8-2

33. Stopcock adapter with 14/20 joints, Quark Enterprises, QA 1420T

34. Vacuum Take-Off, Quark Enterprises, QA-100, with 14/20 outer, 24/40 inner with drip tube

35. 500 mL Erlenmeyer flask with 24/40 joint, Quark Enterprises, QF-6-18

36. MicroVap LV Concentration WorkStation, Caliper Life Sciences

37. Graduated cylinders – Class A, assorted sizes

38. 1-L glass bottle

39. 1-L amber glass bottle

40. Glass fiber filter paper, type GF/C or 934-AH

41. Concentrator tube, 25mm x 150mm with 24/40 joint and tapered bottom, Quark Enterprises, QCT-2440, or equivalent.

42. Syringes, Hamilton gastight, assorted sizes

43. Beakers, assorted sizes

44. Burette clamps

45. Glass funnel with 24/40 joint

46. Glass funnel with 14/20 joint

47. 7/16” stainless steel screen disc, TWP Corporation

48. Glass fiber filter, 13 mm, Sterlitech Inc, GB100R13MM or equivalent

49. Measuring spoon set, stainless steel, ¼, ½, 1 teaspoon, 1 tablespoon

50. GC vial insert, glass, 250 µl, Restek, 21776

51. GC vial Fisher C4010-1

52. 3-ball Snyder columns for KD concentration.

53. Chromacol GC vial, 1.1 mL Screw top with tapered tip, Chromatography Research Supplies, Inc., Item No. 123275

54. 40 mL glass vial with Teflon lined cap

55. Glass wool

56. Sonic bath, Fisher Scientific FS110 or equivalent.

57. GC Cial Caps Fisher C4010-60A

58. Chromacol GC Vial Cap Chromatography Research Supplies, Inc., 309525.

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or in pressure resistant steel drums. Solvents, acids and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer’s storage conditions. See *T-HRMS-WI9446* for the storage and stability of the spiking solutions listed below.

1. Methylene chloride – Pesticide grade or equivalent

2. Hexane – Pesticide grade or equivalent

3. Acetone – Pesticide grade or equivalent

4. Nonane – Aeros Organics, 99%, No. 129115000 or equivalent

5. Methanol – Fisher Scientific, A454, Optima grade or equivalent

6. Silica Gel, 63-200 micron particles, Fisher Scientific, 05-719-822, or equivalent

7. Sulfuric Acid (H_2SO_4), concentrated, Reagent Grade

8. Sodium Hydroxide (NaOH), Reagent Grade

9. Silver Nitrate ($AgNO_3$), Reagent Grade

10. Alumina Basic Super 1, Sorbent Technologies, Cat. # 15160-05

11. Sodium sulfate ($NaSO_4$) – Reagent grade or equivalent.

12. Labeled Compound Spiking Solutions – See *T-HRMS-WI9446*

13. Native Compound Spiking Solutions – See *T-HRMS-WI9446*

14. Cleanup Standard Spiking Solution - See *T-HRMS-WI9446*

15. Percent solids comparison reference

Calibration
Not applicable for this procedure

Preparation of Glassware

See *T-HRMS-WI9485*.

Procedure

A. Preparation of Media:

All media is stored in a desiccator cabinet at room temperature for up to 1 year.

1. Clean sodium sulfate (Na_2SO_4)

a. Load approximately 1250 g of sodium sulfate into a column adsorbent preparation tube. Secure Na_2SO_4 with glass wool packing.

b. Move the tube to the solvent flushing station.

c. With the adsorbent preparation tube in an upright position, attach it to the 4 L filter flask and attach vacuum supply line. Place a 2000 mL solvent bulb on top of the adsorbent preparation tube

d. Add 1500 mL of hexane to the solvent reservoir, when hexane is through the Na_2SO_4 pull dry with vacuum.

e. Detach the 2000 mL solvent reservoir; detach the adsorbent preparation tube from the 4 L filter flask.

f. Insert the adsorbent preparation tube into the tube furnace and attach the tube adapter with the N_2 supply.

g. Start the N_2 supply at 5 to 7 SCFH (standard cubic feet per hour), bake at 225 C in the tube furnace for a minimum of 2 hours with a stream of nitrogen flowing through the tube

h. Turn off the power to the tube furnace and allow the Na_2SO_4 to cool

i. Transfer the Na_2SO_4 from the adsorbent preparation tube to a 1 gallon glass bottle. Tightly close the lid on the glass bottle and store in a desiccator cabinet until further use.

Store in glass jar in desiccator cabinet

2. Clean bulk silica gel.

a. Add 350 g of silica gel to the column adsorbent preparation tube. Secure silica gel with glass wool packing.

b. Move the tube to the solvent flushing station.

c. With the adsorbent preparation tube in an upright position, attach it to the 4 L filter flask and attach vacuum supply line. Place 2000 mL solvent bulb on top of the adsorbent preparation tube.

d. Add 1500 mL of methanol to the solvent reservoir. Gently apply vacuum and draw the methanol through the silica gel.

e. As the surface of the methanol approaches the top surface of the silica gel, add a small portion of 1500 mL of methylene chloride to the solvent reservoir. Allow the methylene chloride to be drawn into the column and then add the rest of the methylene chloride.

f. When all the methylene chloride has been drawn into the adsorbent preparation tube, continue to draw air through the tube for an additional 15-20 minutes using vacuum to pull solvent out of the column until there is very little solvent flow exiting the column.

g. Detach the 2000 mL solvent reservoir; detach the adsorbent preparation tube from the 4 L filter flask.

h. Insert the adsorbent preparation tube into the tube furnace and attach the tube adapter with the N2 supply.

i. Start the N2 supply at 5 to 7 SCFH (standard cubic feet per hour) and turn on the tube furnace to 180°C. Heat the silica gel for 1 hour.

j. Turn off the power to the tube furnace and allow the silica gel to cool.

k. Transfer the silica gel from the adsorbent preparation tube to a 1 gallon glass bottle. Tightly close the lid on the glass bottle and store in a desiccator cabinet until further use.

3. Prepare acid silica gel.

a. Weigh out 800 g of the cleaned silica gel into a 1 gallon glass bottle.

b. Weigh out 352 g of sulfuric acid into a 500 mL beaker.

c. Pour portions of the sulfuric acid from the beaker into the silica gel. Frequently shake and mix the silica gel to break up any clumps that form.

d. Transfer a portion of the silica gel back to the 500 mL beaker to soak up any sulfuric acid that remains in the beaker and then transfer back to the 1 gallon glass bottle.

e. Place the 1 gallon glass bottle on the bottle roller mixer for several hours or until the silica gel is uniform and free flowing.

f. Store the acid silica gel in the tightly closed 1 gallon glass bottle in a desiccator.

4. Prepare basic silica gel.

a. Weigh out 300 g of cleaned silica gel and transfer to a 1 quart glass bottle.

b. Prepare 1N sodium hydroxide by dissolving 4 g of sodium hydroxide in 100 mL of water.

c. Measure out 90 g of 1N sodium hydroxide. Pour portions of the 1N sodium hydroxide into the silica gel. Frequently shake and mix the silica gel to break up any clumps that form.

d. Place the 1 quart glass bottle on the bottle roller mixer for several hours or until the silica gel is uniform and free flowing.

e. Store the basic silica gel in the tightly closed 1 quart glass bottle in a desiccator.

5. Prepare 10% AgNO3 silica gel. The preparation should be done in a manner that minimizes direct exposure to light.

a. Weigh out 350 g of cleaned silica gel into a 1 liter amber glass bottle

b. Prepare an $AgNO3_3$ solution by dissolving 38.89 g of $AgNO3_3$ in 150 mL of water. Be careful to ensure that the AgNO3 is completely dissolved. Sonication/stirring of the solution can be used if necessary.

c. Pour portions of the $AgNO3_3$ solution into the silica gel. Frequently shake and mix the silica gel to break up any clumps that form.

d. Place the 1 L amber glass bottle containing the $AgNO3_3$ treated silica gel on the bottle roller mixer for several hours or until the silica gel is uniform and free flowing.

e. Load the $AgNO3_3$ silica gel into the adsorbent preparation tube, secure with glass wool packing, and insert tube into tube furnace.

f. Attach tube adapter with N_2 supply and purge tube at 5 to 7 SCFH for 30 minutes with the oven lid closed.

g. Turn on power to tube furnace and set temperature to 120C. Purge and heat the $AgNO3_3$ silica gel for at least 16 hours or overnight to activate it.

h. Turn off the power to the tube furnace and allow the $AgNO3_3$ silica gel to cool under N_2 purge.

i. Examine the color of the $AgNO3_3$ silica gel. It should be a cream to light tan color. If the $AgNO3_3$ is a darker brown or gray, it must be discarded and cannot be used.

j. After the $AgNO3_3$ silica gel is cool, transfer back to the 1 L amber glass bottle. Store in desiccator shielded from direct sunlight or room light.

B. Sample preparation

1. Prepare Working Native, Isotopically Labeled, and Cleanup Standard Spiking Solutions as indicated in *T-HRMS-WI9446*.

2. Prepare water sample for extraction

a. Open parallax enter batch scheduling, click on the PE icon, and find the corresponding batch number. Match the sample bottle to the sample on the screen, make sure reading one box is highlighted for the sample number, and press F3 to record sample weightto the table. Continue entering the weight of the full bottle for each sample on the batch. For the blank and OPR enter a weight of 1.5 for reading one and .5 for reading two. Enter all otherinformation into the tables under the different tabs, note anything about the water in the comment section, andsave before you exit the table.

b. Prepare Method Blank and OPR by placing 1 L of reagent water in each of two clean 1-L glass sample bottles.

c. Spike the samples below the surface of the water according to the following table. Use the appropriate calibrated, gas tight syringe. Prior to spiking each sample, clean the syringe using an aliquot of acetone.

Sample	100 µL of Working Labeled Compound Spiking Solution for Dioxin/furan 1.0 mL of Working Labeled Compound Spiking Solution Spike for PCB congener	100 µL of Working Native Compound Spik Solution for Dioxin/fur 1.0 mL of Working Native Compound Spik Solution for PCB conge
Field Sample	Yes	No
Method Blank	Yes	No
OPR	Yes	Yes

NOTE: If there is zero headspace in a sample bottle because it is completely full, there is no space for the spiking solution. In that event, prior to spiking, remove a few mL of sample from the bottle using a disposable pipette and transfer it to the 2-L sep funnel that will be used for extraction, or transfer it to the Buchner funnel that will be used for filtration.

d. Carefully shake each sample bottle and allow the spikes to equilibrate for a minimum of 1 hour. Sonicate the water sample for 5 minutes.

e. If the sample has less than 1% solids, determined by a comparison with a reference, pour the contents of the sample bottle into a 2-L separatory funnel. If visible particulates are present, rinse the bottle twice with approximately 5 mL of reagent water and add to the separatory funnel.

NOTE: The % solids comparison reference is a 1L amber bottle containing 10.00g soil and 1000ml DI water. The preparation is logged into a reagent logbook and is given a reagent lot number.

f. Rinse the sample bottle with methylene chloride and transfer rinse to 2-L separatory funnel that contains the sample.

g. For samples that have greater than 1% solids, filter the sample using vacuum filtration through a Buchner funnel and two glass fiber filter papers, placed on top of a metal screen, into a 2 -L filter flask.

NOTE: Most of any CDD/CDF present in the water sample will be adsorbed on to particulate matter that is present in the sample.

Be very careful to rinse all particulate from the sample bottle by swirling the bottle during the transfer followed by a minimum of 2 small quantity rinses of reagent water. Rinse the sides of the Buchner funnel with small quantities of reagent water so that all particulates are positioned on top of the filter paper. Take a small quantity of methylene chloride and rinse the filtration apparatus into the 2-L filtration flask containing the filtrate.

h. Remove the filter paper and place into a glass fiber thimble. Extract the filter paper and particulates using an SDS extraction apparatus as described in Analysis #11030, 13234.

i. Transfer the filtrate to a 2-L separatory funnel. Use small quantities of reagent water to rinse the filtrate flask and add rinsate to the 2-L separatory funnel.

3. Extract water sample

a. Using a solvent pump, measure 60 mL of methylene chloride to the sample bottle; shake the bottle for at least 60 seconds, then transfer the solvent to the 2-L separatory funnel.

b. Stopper the funnel, invert it, and vent immediately. Handshake and vent frequently until the pressure is stable. Place the sample on the automatic shaker and shake at the designated speed (approx. 145 rpm) for 2 minutes with the stopcocks closed.

c. Place the separatory funnel on the rack and allow it to sit undisturbed for 10 minutes. This allows the organic and aqueous layers to separate. If an emulsion forms and is> 1/3 of the volume of the solvent layer, mechanical techniques such as stirring, the use of a miniature separatory funnel, or centrifugation must be employed to complete the separation.

d. Wet the sodium sulfate in the column with approximately 20 mL of methylene chloride. Open the 2-L separatory funnel stopcock and drain the methylene chloride solvent layer into a 250 mL glass beaker. Transfer the solvent from the beaker through a sodium sulfate column that is

approximately ½ full of sodium sulfate that is attached to a K-D apparatus containing a few Teflon boiling chips. Rinse the beaker with approximately 10 mL of methylene chloride and transfer rinsings to K-D.

e. Using a solvent pump add 60 mL of methylene chloride to the 2-L separatory funnel and repeat steps b through d, venting only as necessary.

f. Repeat steps b through d to perform a third extraction cycle. Drain the extracted water sample to waste. Rinse the 2-L separatory funnel with approximately 20 mL of methylene chloride and transfer through sodium sulfate to K-D concentrator. Repeat the rinse of the separatory funnel twice. Using a wash bottle, rinse the sodium sulfate in the sodium sulfate column with 2 rinses of approximately 20 mL of methylene chloride.

g. Attach a 3-ball Snyder column to the K-D, and concentrate the extract on a steam bath at 90°to 95°C to 5 to 10 mL. Exchange the extract to hexane by carefully adding 50 mL of hexane through the 3-ball Snyder column. Concentrate to 5 to 10 mL. Allow the sample to cool for approximately 10 minutes.

h. For samples with particulate SDS extracts, add the KD extract to the SDS toluene extract in the concentrator tube prior to dilution with hexane. (See SDS extraction Analysis #11030, 13234).

i. The resulting extract is then ready for column cleanup.

j. Open the prep entry tab in parallax and find your batch in the table. Match the bottle with the sample number and highlight the reading two box, press F3 to record the weight of the empty bottle. When all samples have both weights entered press calculate to get the sample weight.

k. If the sample is a drinking water scheduled for analysis 12935, and the extract is colorless and clear, extract cleanup is not required. For these extracts, replace the K-D with a 2-ball Snyder column, secure the connection with a blue clip and concentrate on a steam bath at 90° to 95°C to <1 mL. Allow to cool for approximately 5 minutes. Using a disposable pipet, transfer the extract quantitatively, with hexane, into a Chromacol vial.

C. Extract Cleanup Procedure

The cleanup of sample extracts is needed to remove potential interferences that are observed in the HRMS analysis. Different combinations of adsorbents can be used to achieve cleanup goals and separate analytes into fractions. Routine extract cleanups require the following adsorbent column combinations:

Analyte	Adsorbent
Dioxin/furans	Multi-layer silica gel + Alumina
PCB Congeners	Multilayer silica gel

NOTE: Based on the severity of the sample matrix and the concentrations of PCBs, alumina can also be used in addition to the multilayer silica gel for the clean-up of PCB congener extracts.

NOTE: Cleanup sorbents may be modified based on client specific requirements and a vetting of the performance of the modified sorbent.

1. Prepare the alumina column

a. Place a 7/16” diameter stainless steel screen disc in the bottom of the column and place a 13 mm glass fiber filter paper on top of the screen.

b. In a hood, place Cleanup Column #2 in a buret clamp so it can be loaded with adsorbent. Place a glass funnel with a 14/20 joint on top of Cleanup Column #2.

c. Add 1 ½ teaspoons (5.5 g) of alumina to the column, pack the column. The measurement of the adsorbent requires a spoon full of media to be scraped with one pass of a spatula to make sure the measurement is reproducible and level full.

d. Add Na_2SO_4 to the top of the alumina in the column until the level of the Na_2SO_4 reaches the flare point of the threaded column fitting on the column top, or 1/2 teaspoon if using the DF column set.

e. Attach to the flask, clip to the stopcock, and cover with hexane.

2. Prepare multi-layer silica gel column

a. Place a 7/16” diameter stainless steel screen disc in the bottom of the column and a 13 mm glass fiber filter on top of the screen. Fit the column with a Teflon/silicon seal ring, Teflon side down, and a red cap with hole, the cap and ring are not needed for the DF column set.

b. In a hood, place Cleanup Column #1 in a burette clamp so that it can be filled with adsorbent. Place a glass funnel with a 24/40 joint on top of the column to facilitate filling. The silica gel tends to create small amounts of airborne dust so the filling operation must be done in a hood.

c. Add layers of silica gel according to the following table. The measurement of the adsorbent requires a spoon full of media to be scraped with one pass of a spatula to make sure the measurement is reproducible and level full.

Layer position	Material	Measurement	Weight (g)
Top	Na_2SO_4	½ teaspoon	5.0
	Acid Silica Gel	1 tablespoons	7.2
	Silica Gel	¼ teaspoon	0.5
	Basic Silica Gel	1 teaspoon	2.5
	Silica Gel	¼ teaspoon	0.5
	$AgNO3_3$ Silica Gel (1)	½ teaspoon	1.2
Bottom	Silica Gel (1)	¼ teaspoon	0.5

NOTE: Omit the $AgNO3_3$ and Silica Gel segment of the column when performing cleanup of PCB Congener extracts.

3. Assemble column set

Assemble the column cleanup set by attaching the glassware pieces to one another in the following order (top to bottom) and clamping the set in a burette clamp at the column cleanup workstation:

a. 250 mL solvent reservoir

b. Cleanup Column #1 multi-layer silica gel column

c. Cleanup Column #2 alumina column

d. stopcock adapter

e. vacuum adapter

f. 500 mL filter flask with vacuum supply line

g. pre-wet the column with approximately 20 mL of hexane.

4. Condition the columns

a. Condition the column by eluting with 50 mL of hexane. Discard the hexane and rinse the flask with methylene chloride, acetone, and ending with hexane, and reattach the flask to the column apparatus at this point for congener cleanups. If conditioning the column for DF cleanups, discard hexane when the cleanup is complete.

b. If necessary, detach the silica gel column from the alumina column and add hexane to fill the headspace above the alumina column. Reattach the silica gel column to the alumina column.

All samples for D/F, with the exception of clear and colorless drinking water extracts for TCDD only, will require the multi-layer silica gel-alumina column described above. All samples for PCBs will require the multi-layer silica gel column.

5. Cleanup of Dioxin/Furan Extracts:

When using the multi-layer Silica Gel with the Alumina Column for dioxin/furan cleanup, proceed in the following manner. All samples, with the exception of clear and colorless drinking water extracts for TCDD only, will require the multi-layer silica gel-alumina column described above.

a. If necessary, detach the silica gel column from the alumina column and add hexane to fill the headspace above the alumina column. Reattach the silica gel column to the alumina column, remove the solvent reservoir.

b. Add 100 µl for Dioxin/Furan, 1.0mL for PCB congeners of working cleanup standard solution to sample extract. Dioxin/furans and PCBs each have their corresponding cleanup solutions.

c. Using hexane, quantitatively transfer the extract from the ampule/concentrator tube to the head of the silica gel column.

d. Start the column flow to allow the sample extract to flow into the silica gel column, vacuum can be used as necessary to assist in the adjustment of the column flow.

e. Add a few milliliters of hexane using a Teflon solvent bottle to rinse the extract and the cleanup standard spike into the column adsorbent, attach the solvent reservoir.

f. Add 50 mL of hexane to the solvent reservoir and elute the column at about 2-3 drops per second.

g. Stop the column flow as the hexane surface approaches the top of the alumina layer. At this point, the silica gel column will have gone “dry”.

h. Remove the silica gel column and turn off the vacuum to the column, allow the hexane on alumina column to elute to waste.

i. Elute, by gravity, the alumina column with 25 mL of 5% methylene chloride in hexane. This fraction is discarded.

j. Detach the 500 mL filter flask and attach the appropriately labeled concentrator tube. Elute, by gravity, the alumina column with 25 mL of methylene chloride and collect this fraction containing the PCDD/PCDF compounds in the concentrator tube.

k. Detach the concentrator tube from the column set and place in MicroVap concentrator. Set the water bath temperature to 40°C and the N_2 flow to about 5 psi. Concentrate the extract to approximately 100-200 µl and quantitatively transfer to a GC vial with insert.

l. Submit the concentrated extract in the vial to the high resolution mass spectrometry lab for PCDD/PCDF analysis.

6. Cleanup of PCB Congener Extracts:

When using the multi-layer Silica Gel column for PCB Congeners, proceed in the following manner.

a. Condition the column by eluting with 50 mLs of hexane into a 250 mL or 500 mL filter Erlenmeyer flask. Discard the hexane and rinse the filter flask with the following solvents, always ending with hexane: methylene chloride, acetone and hexane.

b. Add 1.0mL of working cleanup standard solution to sample extract. Dioxin/furans and PCBs each have their corresponding cleanup solutions.

c. Using hexane, quantitatively transfer the extract from the concentrator tube to the head of the silica gel column.

d. Start the column flow to allow the sample extract to flow into the silica gel column, under vacuum at a rate of 2-3 drops per second.

e. Add a few milliliters of hexane using a Teflon solvent bottle to rinse the extract and the cleanup standard spike into the column adsorbent, attach the solvent reservoir.

f. Load 50 mL of hexane and elute the column at 2 to 3 drops per second, using vacuum as needed.

g. Collect the entire 50 mL hexane eluent.

h. Open stopcock slowly maintaining the 2-3 drop per second drip rate as the column starts to dry. Stop the elution as the silica gel column goes dry.

i. Detach the silica gel column.

j. Add 20 µL nonane to each flask. Divide the hexane eluent evenly between three concentrator tubes. Place tubes in MicroVap concentrator. Set the water bath temperature to 40°C and the N_2 flow to about 5 psi. Concentrate the extracts far enough so that they can be combined into a single tube for each sample. Concentrate each single tube to <1 mL and quantitatively transfer to a Chromacol vial. Submit the concentrated extract in the vial to the high resolution mass spectrometry lab for PCB analysis.

Calculations

See analysis method.

Statistical Information/Method Performance

See analysis method.

Quality Assurance/Quality Control

All samples are spiked with ^{13}C labeled compounds to monitor analyte recovery using isotope dilution quantitation. A sample “batch” will consist of no more than 20 field samples per set of

Quality Control (QC) samples. QC samples will consist of a method blank (reagent water), and an Ongoing Performance and Recovery (OPR) sample (reagent water blank spiked with all compounds to be determined and carried through the entire procedure). See Analysis #10915, 11031, 11645, 11650, 12935, 12936, 12937, 13232, 13233 and analysis #12154, 12429, 13707, 13708 for acceptance criteria and corrective action. If any client, state, or agency has more stringent QC or batch requirements, these must be followed instead.

T-HRMS-WI9432 PCB Congeners by Method 1668 HRGC/HRMS in Aqueous and Solid Matrices
T-HRMS-WI9446 Standards Management in the High Resolution Mass Spectrometry Laboratory
T-HRMS-WI9476 Determination of Tetra- Through Octa- Chlorinated Dioxins and Furans in water/solid/food/feed samples using HRGC/HRMS by EPA 1613B or SW-846 Method 8290A
T-HRMS-WI9485 Glassware Cleaning for HRMS Extractions
T-HRMS-WI9488 Soxhlet Extraction Procedure for HRMS Analysis in a Solid Matrix by Methods: 1613B, 8290A, 1668C, and 1668A

Attachment:
Attachment I

End of document

Version history

Version	Approval	Revision information
2	24.OCT.2016	
3	18.JUN.2018	
4	05.DEC.2018	

Attachment I

Analysis # 14069 (DRBC Congeners in Water-SepF) requirements for extraction.

A. Sample preparation

NOTE: 2L of sample is used for this extraction. It may be 2 1L bottles, or a 2L bottle. If sample is submitted in on large container, a graduated cylinder is used to transfer the sample aliquots to the 2L separatory funnel for extraction. Each liter aliquot of sample is extracted separately and combined prior to starting the final cleanup steps.

1. Prepare Working Native, Isotopically Labeled, and Cleanup Spiking Solutions as indicated in T-HRMS-WI9446.

2. Prepare water sample for extraction

   a. Open parallax enter batch scheduling, click on the PE icon, and find the corresponding batch number. Match the sample bottle to the sample on the screen, make sure reading one box is highlighted for the sample number, and press F3 to record sample weight to the table. Continue entering the weight of the full bottle for each sample on the batch. For the blank and OPR enter a weight of 1.5 for reading one and .5 for reading two. Enter all other information into the tables under the different tabs, note anything about the water in the comment section as well as the 2nd bottle code used for extraction if more than one bottle was used, and save before you exit the table.

   b. Prepare Method Blank by placing 1 L of reagent water in each of two clean 1-L glass sample bottles. Prepare OPR by placing 1 L of reagent water in each of two clean 1-L glass sample bottles. Prep data should have the sum volume of the two 1L bottles used for the extraction.

   c. Spike the samples below the surface of the water according to the following table. Use the appropriate calibrated, gas tight syringe. Prior to spiking each sample aliquot, clean the syringe using an aliquot of acetone.

Sample	0.5 mL of Working Labeled Compound Spiking Solution Spike for PCB congener	0.5 mL of Working Native Compound Spiking Solution for PCB congener
Field Sample	Yes	No
Method Blank	Yes	No
OPR	Yes	Yes

NOTE: If there is zero headspace in a sample bottle because it is completely full, there is no space for the spiking solution. In that event, prior to spiking, remove a few mL of sample from the bottle using a disposable pipette and transfer it to the 2-L separatory funnel that will be used for extraction, or transfer it to the Buchner funnel that will be used for filtration.

d. Carefully shake each sample bottle and allow the spikes to equilibrate for a minimum of 1 hour. Sonicate the water sample for 5 minutes.

e. If the sample has less than 1% solids, determined by a comparison with a reference, pour the contents of the sample bottle into a 2-L separatory funnel. If visible particulates are present, rinse the bottle twice with approximately 5 mL of reagent water and add to the separatory funnel.

NOTE: The % solids comparison reference is a 1L amber bottle containing 10.00g soil and 1000ml DI water. The preparation is logged into a reagent logbook and is given a reagent lot number.

f. Rinse the sample bottle with methylene chloride and transfer rinse to 2-L separatory funnel that contains the sample.

g. For samples that have greater than 1% solids, see Procedure B. 2.e.- I of the main document.

3. Extract water sample

a. Using a solvent pump, measure 60 mL of methylene chloride to the sample bottle; shake the bottle for at least 60 seconds, then transfer the solvent to the 2-L separatory funnel.

b. Stopper the funnel, invert it, and vent immediately. Handshake and vent frequently until the pressure is stable. Place the sample on the automatic shaker and shake at the designated speed (approx. 145 rpm) for 2 minutes with the stopcocks closed.

c. Place the separatory funnel on the rack and allow it to sit undisturbed for 10 minutes. This allows the organic and aqueous layers to separate. If an emulsion forms and is >1/3 of the volume of the solvent layer, mechanical techniques such as

stirring, the use of a miniature separatory funnel, or centrifugation must be employed to complete the separation.

d. Wet the sodium sulfate in the column with approximately 20 mL of methylene chloride. Open the 2-L separatory funnel stopcock and drain the methylene chloride solvent layer into a 250 mL glass beaker. Transfer the solvent from the beaker through a sodium sulfate column that is approximately ½ full of sodium sulfate that is attached to a K-D apparatus containing a few Teflon boiling chips. Rinse the beaker with approximately 10 mL of methylene chloride and transfer rinsings to K-D.

e. Using a solvent pump add 60 mL of methylene chloride to the 2-L separatory funnel and repeat steps b through d, venting only as necessary.

f. Repeat steps b through d to perform a third extraction cycle. Drain the extracted water sample to waste. Rinse the 2-L separatory funnel with approximately 20 mL of methylene chloride and transfer through sodium sulfate to K-D concentrator. Repeat the rinse of the separatory funnel twice. Using a wash bottle, rinse the sodium sulfate in the sodium sulfate column with 2 rinses of approximately 20 mL of methylene chloride.

g. Attach a 3-ball Snyder column to the K-D, and concentrate the extract on a steam bath at 90°to 95°C to 5 to 10 mL. Exchange the extract to hexane by carefully adding 50 mL of hexane through the 3-ball Snyder column. Concentrate to 10 mL. Allow the sample to cool for approximately 10 minutes.

h. For samples with particulate SDS extracts, add the KD extract to the SDS toluene extract in the concentrator tube prior to dilution with hexane. (See SDS extraction Analysis #11030, 13234).

i. The resulting extract is then ready for column cleanup.

j. Open the prep entry tab in parallax and find your batch in the table. Match the bottle with the sample number and highlight the reading two box, press F3 to record the weight of the empty bottle. When all samples have both weights entered press calculate to get the sample weight.

C. Extract Cleanup Procedure

The cleanup of sample extracts is needed to remove potential interferences that are observed in the HRMS analysis. Different combinations of adsorbents can be used to achieve cleanup goals and separate analytes into fractions. Routine extract cleanups require the following adsorbent column combinations:

Analyte	Adsorbent
PCB Congeners	Multilayer silica gel

NOTE: Based on the severity of the sample matrix and the concentrations of PCBs, alumina can also be used in addition to the multilayer silica gel for the clean-up of PCB congener extracts.

NOTE: Cleanup sorbents may be modified based on client specific requirements and a vetting of the performance of the modified sorbent.

2. Prepare multi-layer silica gel column

a. Place a 7/16” diameter stainless steel screen disc in the bottom of the column and a 13 mm glass fiber filter on top of the screen. Fit the column with a Teflon/silicon seal ring, Teflon side down, and a red cap with hole, the cap and ring are not needed for the DF column set.

b. In a hood, place Cleanup Column #1 in a burette clamp so that it can be filled with adsorbent. Place a glass funnel with a 24/40 joint on top of the column to facilitate filling. The silica gel tends to create small amounts of airborne dust so the filling operation must be done in a hood.

c. Add layers of silica gel according to the following table. The measurement of the adsorbent requires a spoon full of media to be scraped with one pass of a spatula to make sure the measurement is reproducible and level full.

Layer position	Material	Measurement	Weight (g)
Top	Na_2SO_4	½ teaspoon	5.0
	Acid Silica Gel	1 tablespoons	7.2
	Silica Gel	¼ teaspoon	0.5
	Basic Silica Gel	1 teaspoon	2.5
	Silica Gel	¼ teaspoon	0.5
	$AgNO3_3$ Silica Gel (1)	½ teaspoon	1.2
Bottom	Silica Gel (1)	¼ teaspoon	0.5

NOTE: Omit the $AgNO3_3$ and Silica Gel segment of the column when performing cleanup of PCB Congener extracts.

3. Assemble column set

Assemble the column cleanup set by attaching the glassware pieces to one another in the following order (top to bottom) and clamping the set in a burette clamp at the column cleanup workstation:

a. 250 mL solvent reservoir

b. Cleanup Column #1 multi-layer silica gel column

c. stopcock adapter

d. vacuum adapter

e. 500 mL filter flask with vacuum supply line

f. pre-wet the column with approximately 20 mL of hexane.

4. Condition the columns

a. Condition the column by eluting with 50 mL of hexane. Discard the hexane and rinse the flask with methylene chloride, acetone, and ending with hexane, and reattach the flask to the column apparatus at this point for congener cleanups.

All samples for PCBs will require the multi-layer silica gel column.

5. Cleanup of PCB Congener Extracts:

When using the multi-layer Silica Gel column for PCB Congeners, proceed in the following manner.

a. Condition the column by eluting with 50 mLs of hexane into a 250 mL or 500 mL filter Erlenmeyer flask. Discard the hexane and rinse the filter flask with the following solvents, always ending with hexane: methylene chloride, acetone and hexane.

b. Add 1.0 mL of working cleanup standard solution to only one of the aliquots of sample extract.

c. Using hexane, quantitatively transfer the extract from both of the aliquots of sample extract ampoules for each respective sample to the head of the silica gel column.

d. Start the column flow to allow the sample extract to flow into the silica gel column, under vacuum at a rate of 2-3 drops per second.

e. Add a few milliliters of hexane using a Teflon solvent bottle to rinse the extract and the cleanup standard spike into the column adsorbent, attach the solvent reservoir.

f. Load 50 mL of hexane and elute the column at 2 to 3 drops per second, using vacuum as needed.

g. Collect the entire 50 mL hexane eluent.

h. Open stopcock slowly maintaining the 2-3 drop per second drip rate as the column starts to dry.Stop the elution as the silica gel column goes dry.

i. Detach the silica gel column.

j. Add 20 µL nonane to each flask. Divide the hexane eluent evenly between three concentrator tubes. Place tubes in MicroVap concentrator. Set the water bath temperature to 40°C and the N_2 flow to about 5 psi. Concentrate the extracts far enough so that they can be combined into a single tube for each sample. Concentrate each single tube to< 1 mL and quantitatively transfer to a Chromacol vial. Submit the concentrated extract in the vial to the high resolution mass spectrometry lab for PCB analysis.

E-18

PCB Congeners by Method 1668 HRGC/HRMS in Aqueous and Solid Matrices
(Rev. 7; 05/30/2018; T-HRMS-WI9432)

LIMS ID
Analysis 12154, 12429, 13584, 13707, 13708

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Revision Log

Revision: 7		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reference not required	Removed QA-QP11176. Reference to Level 1 Document not needed.
Reagents and Standards	Clarification	Added more detail for storage conditions and management of expiration dates
Procedure E	Reflects current process	Added language regarding detections in sample data that exceed linear range of instrumentation removed references to 1-P-QM-PRO-9015510
Procedure F	Reflects current process	Resolution check procedure was updated by ThermoScientific to provide better documentation of instrument tuning to satisfy method requirements
Statistical Information/Method Performance	Reflects current practices	Revised entire section to match current EPA requirements.
Cross reference	reflect current practice	deleted 1-P_QM-PRO-90115510 and added *QA-SOP11892*

Revision: 6	**Effective Date:** 27-JAN-2016	
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Correction	Revised title to edit the word matrices
Historical/Local Document Number	New LIMS analysis scans	Added Analysis #13707 and 13708
Safety Precautions and Waste Handling	Reflects current process	Added the word local in reference to accumulating, managing, and disposing laboratory waste
Personnel Training and Qualifications	Reflects current process	Added language regarding IDOCs and DOCs
Sample Collection, Preservation and Handling	Revised in response to Agency audit	Clarified storage of tissue extracts and use of amber glass containers for sample collection.
Procedure D.2	Revised in response to Agency audit	Added section regarding ICV analysis.
Procedure F.1	Revised in response to Agency audit	Added statement about which lock masses are being used to document mass resolution for each descriptor.

Reference

1. EPA Method 1668A, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, August 2003

2. EPA Method 1668C, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, April 2010

3. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #10914	Separatory Funnel Sample Extraction Procedure for HRMS Analysis in a Water Matrix
Analysis #11030	Soxhlet Sample Extraction Procedure for HRMS Analysis in a Solid Matrix
T-HRMS-WI19229	DFS HRGC/HRMS Preventative and Corrective Maintenance
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
T-HRMS-WI9433	Processing High Resolution Mass Spectrometry Data using TargetQuan
T-HRMS-WI9446	Standards Management in the High Resolution Mass Spectrometry Laboratory

Purpose

This procedure describes the setup and calibration of a HRGC/HRMS (high resolution capillary column gas chromatography/ high resolution mass spectrometry) system to acquire sample data to identify and quantitate the PCB congeners in various matrices.

Scope

This method is for the determination of PCB congeners in water, soil, sediment, sludge, tissue, and other sample matrices using isotope dilution quantitation by high resolution gas chromatography/high resolution mass spectrometry (HRGC/ HRMS). The method can be used for the analysis of 209 PCB congeners, or selected sublists of PCB congeners. The preparation and cleanup of the sample extracts is described in SOPs listed in Cross References.

Basic Principles

After sample extraction and cleanup, the extract is concentrated to near dryness. Immediately prior to injection, internal standards are added to each extract, and an aliquot of the extract is injected into the gas chromatograph (GC). The analytes are separated by the GC and detected by a high-

resolution (≥10,000) mass spectrometer. Two exact m/z's are monitored for each analyte. An individual PCB congener is identified by comparing the GC retention time and ion abundance ratio of two exact m/z's with the corresponding retention time of an authentic standard and the theoretical or acquired ion-abundance ratio of the two exact m/z's. Quantitative analysis is performed using selected ion current profile (SICP) areas, in one of three ways:

1. For the native PCB congeners, the GC/MS system is calibrated using a multi-point calibration and the concentration of each compound is determined using the isotope dilution technique.

2. When only the Tox/LOC congeners are multi-point calibrated, a single-point calibration is employed for all other non-labeled congeners, and quantitation is performed using the internal standard technique. The internal standards are the labeled congeners spiked into the sample, thus affording recovery correction for all congeners.

3. For $^{13}C_{12}$ labeled compounds, the GC/MS system is calibrated using a multi-point calibration and the concentration of each compound is determined using the internal standard technique.

The quality of the analysis is assured through reproducible calibration and testing of the extraction, cleanup, and GC/MS systems.

Definitions

1. PCDD - Polychlorinated dibenzodioxin
2. PCDF - Polychlorinated dibenzofuran
3. GC - Gas Chromatography
4. HRMS - High Resolution Mass Spectrometry
5. MID - Multiple Ion Detection scan mode on the Thermo DFS HRMS system
6. m/z – mass to charge ratio
7. OPR - Ongoing Performance and Recovery spike
8. PCB - Polychlorinated Biphenyl
9. PCB congener - Single, unique, well-defined chemical compound in the PCB category
10. PCB homolog - PCB congeners with equal numbers of chlorine substituents

11. VER - Calibration Verification standard

12. DFS – ThermoScientific DFS High Resolution Mass Spectrometer

13. LOC – Level of chlorination

Interference

Method interferences may be caused by impurities in solvents, reagents, and glassware, or other hardware used in the processing of samples. All glassware is solvent rinsed before use and a method blank is performed with each extraction batch to demonstrate that potential contamination of the extraction and analysis system is in control.

Coextracted organics such as PCDD/PCDFs, polychlorinated diphenyl ethers, polynuclear aromatics, and organochlorine pesticides will also contribute to analysis interference. Post extraction cleanups should eliminate or sufficiently reduce these interferences to allow reliable determination of PCB congeners.

Interferences may pose a problem in the determination of congeners 81, 123, 126, and 169 in some samples with high concentrations of highly chlorinated congeners. The loss of one or more chlorines from a closely eluting higher chlorination congener may inflate or produce a false concentration for a less-chlorinated congener. Data must be examined for interferences from closely eluting congeners.

Safety Precautions and Waste Handling

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound and reagent should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means are available such as fume hoods, safety glasses, lab coats, and gloves. See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. Two waste streams have been defined to handle PCB sample processing waste:

A. Flammable GC Vials w/PCBs – This waste stream consists of sample extracts in GC vials

B. Samples with PCBs – This waste stream consists samples with PCBs greater than 50 ppm.

Expired PCB standards are routinely collected for disposal through a lab pack process and arrangements with the lab waste disposal vendor.

Personnel Training and Qualifications

Education Requirement: Degree in science or relevant experience

Each new chemist will train with an experienced chemist for the first 12 weeks. The first 12 weeks are spent working one−on−one with the trainer. This time may be less if the new chemist has prior experience or relevant analytical chemistry background.

During the training period, the new chemist will learn daily maintenance, column and source changing procedures, calibration techniques, data review, and forms generation. He/she is also required to read all relevant SOPs and EPA methods and must have a completed and documented Initial Demonstration of Capability (i.e., quad study) that yielded acceptable recoveries.

Personnel performing this procedure must have documentation of reading, understanding and agreeing to follow this version of the SOP and must have completed a documented annual Demonstration of Capability.

Sample Collection, Preservation, and Handling

Samples are collected in amber glass containers. Aqueous samples are stored refrigerated at 0° to 6°C, not frozen, in the dark. Aqueous samples are to be tested for residual chlorine and pH upon sample receipt. If residual chlorine is present, add 80 mg of sodium thiosulfate ($Na_2S_2O_3$) per liter of water. If the pH is greater than 9, adjust to pH 7-9 with sulfuric acid. This procedure is performed by the Sample Support Department. Aqueous samples are stored refrigerated at 0 - 6°C, not frozen, in the dark. Solid samples are not preserved and are stored refrigerated at 0° to 6°C, not frozen, in the dark. Tissue samples are frozen (< −10°C) if sample preparation does not begin immediately upon receipt at the laboratory.

There are no demonstrated maximum holding times associated with PCBs in aqueous, solid, semi-solid, tissues, or other sample matrices. Internal operations require that samples are extracted within 30 days of sample collection and analyzed for PCBs within 40 days of extraction. All non-

tissue sample extracts are stored at 0 - 6°C, not frozen, in the dark prior to analysis. If not placed on an instrument for analysis within 24 hours of receipt, tissue sample extracts must be stored in the dark at less than -10°C.

If stored in the dark at < −10°C, sample extracts may be stored for up to one year.

Apparatus and Equipment

1. 10−µL syringe

2. 25−µL syringe

3. Thermo Scientific TRACE GC Ultra gas chromatograph with AS TriPlus Autosampler or equivalent

4. SPB-octyl column: 30 meter X 0.25 mm ID X 0.25 um film thickness

5. Thermo Scientific DFS high resolution mass spectrometer or equivalent

6. Dell Optiplex 960, Core 2 Duo CPU, 3.2 GB Ram computer system, or equivalent

7. ThermoScientific instrumentation software: DFS Instrument Software 2.2.1.717, TargetQuan Programs 3.1 1370, Xcalibur 2.2 or current version.

The preventative and corrective maintenance schedules and procedures associated with the Trace Ultra gas chromatograph and DFS mass spectrometer are outlined in *T-HRMS-WI19229*.

Reagents and Standards

All stocks and standards prepared from vendor supplied ampoules are stored in the dark at room temperature in amber screw-cap vials. See *T-HRMS-WI9446* regarding preparation of working solutions.

Purchased standard stock solutions are supplied with a Certificate of Analysis/Documentation package that describes the mixture compounds and their concentrations. This standard documentation is stored in the Standards – Certificates of Analysis logbook.

Expiration dates are managed through the Standards Maintenance and Tracking (SMT) database. Stock solutions transferred from sealed glass ampoules to screw-top vials are given expiration dates

of 1 year from opening date, or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 1 year from the preparation date, or the earliest expiration date from any stock solution used during preparation. Working native compound spiking solutions, labeled compound spiking solutions, and cleanup standard spiking solutions are given expiration dates of 1 month. Working solutions may be re-certified if they are not consumed by operations prior to expiration date.

1. Perfluorokerosene (PFK) high boiling, Sigma-Aldrich part number 77275 or equivalent. Store at room temperature for up to one year.

2. Nonane, 99%, Acros Organics No. 12911 or equivalent. Store at room temperature for up to one year.

3. Internal Standard Spiking Solution – See *T-HRMS-WI9446*

4. Calibration and Verification Solutions – See *T-HRMS-WI9446*

5. Cleanup Standard Spiking Solution – See *T-HRMS-WI9446*

Calibration

See Procedure D for details on the calibration. In general, CSL, CS1, CS2, CS3, CS4, and CS5 are included in the initial calibration. (See *T-HRMS-WI9446* for calibration concentrations.) Standards that vary in concentration from these mixtures can be used if specific project requirements exist. If the % Relative Standard Deviation (RSD) for the response factors is< 20% for the native compounds, the average response factor can be used. Otherwise, corrective actions must be taken until the % RSD criteria are met. A calibration verification (VER) standard is analyzed every 12 hours. See procedure F and QA/QC section 1 for acceptance criteria.

Procedure

A. References

1. DFS Operating Manual, Revision B - 1195600

2. DFS Hardware Manual, Revision E – 1194840

B. DFS mass calibration and tuning

Tune and calibrate the DFS such that mass resolution is >10,000 (10% valley) and the mass deviation between the exact m/z and the theoretical m/z is less than 5 ppm. Document mass resolution across the range of masses used for PCB congener analysis prior to the start of analysis.

Masses listed in EPA method 1668 include the mass of an electron in the fragment mass. In setting up the DFS, all exact masses configured in the system use masses calculated with the mass of an electron (0.000548u) subtracted from fragment mass.

NOTE: It is desirable to keep the amount of PFK flowing into the ion source as low as possible to minimize the level of interference between the 223.9867 PFK ion with the 223.9969 dichlorobiphenyl M+2 ion.

C. Establishing gas chromatographic conditions and MID descriptors

1. Chromatographic conditions

a. Column - 30 meter X 0.25 mm ID X 0.25 µm film Supelco SPB-octyl column or equivalent.

b. Injector – Split/Splitless in splitless mode. Injector temperature 270°C with vacuum compensated constant carrier flow, splitless time of 1.20 min, and split flow of 70 ml/min.

c. Gas – Helium at approximately 1.0 ml/min, constant flow mode.

d. Transfer line temp - 270°C

e. Oven program – initial temp 100°C for 2 minutes, ramp at 10°C/min to 150°C for 1 minute, then ramp at 2.5°C/min to 270°C, final hold time 2 minutes.

f. Chromatographic conditions may be adjusted to optimize retention times and attain chromatographic resolution specifications. The following requirements must be satisfied:

(1) The retention time for PCB209 is greater than 55 minutes.

(2) The valley between peaks for PCB34 and PCB23 is < 40%.

(3) The valley between peaks for PCB187 and PCB182 is < 40%.

(4) PCB156 and PCB157 coelute with retention times within 2 seconds of each other.

2. Create MID descriptors

a. Use the Xcalibur Instrument Setup to create the PCB congener MID. Typical MID data is shown in Table 3.

b. Inject the PCB 209 Congener Cal Std to determine retention times for the 209 PCB congeners. Alternatively, a set of five standard solutions is available to determine retention times for all 209 PCB congeners and identify which PCBs co-elute on the GC system (refer to *T-HRMS-WI9446*). It is good laboratory practice to run these solutions after installing a new column or setting up analysis on a new instrument as slight variations in chromatographic resolution may occur.

c. Based on the retention time data of the PCB 209 Congener Cal Std, determine the optimal retention times for switching from one MID descriptor to the next MID descriptor. It is critical to select regions of the chromatogram where no PCB congener peaks are eluting. A MID switch takes about 0.10 minutes. Typical retention times for MID switches are shown in Table 2.

D. PCB Congener calibration

There are two ways in which the system may be calibrated for congener analysis. The routine calibration is a six-point calibration for all 209 PCB congeners. Alternatively, the Tox/LOC PCB congeners are multi-point calibrated and the remaining congener response factors are determined by the daily calibration verification standard (single-point calibration). In general, PCB CSL Cal Std, PCB CS1 Cal Std, PCB CS2 Cal Std, PCB CS3 Cal Std, PCB CS4 Cal Std, PCB CS5 Cal Std are included in the initial calibration (See *T-HRMS-WI9446* for calibration concentrations). Standards that vary in concentration from these mixtures can be used if specific project requirements exist.

The multi-point calibration of all 209 PCB congeners includes an additional fourteen labeled compounds that are not noted in EPA 1668A or 1668C. The fourteen additional labeled compounds are used in the average RF for their respective LOC. They are also used to determine RT and RRT of the associated native congeners. When performing the Tox/LOC calibration procedure these additional compounds are not considered.

Native congeners that have an isotopically labeled analog are quantitated using the analog as the retention time and quantitation reference. Native congeners that do not have an isotopically labeled analog use the closest eluting labeled congener, with the same LOC, as the retention time reference. The native congeners that do not have an isotopically labeled analog use an average (mean) response of the labeled compounds at each LOC as the quantitation reference. Retention time and quantitation references are listed in Table 7 (multi-point calibration) and Table 7A (Tox/LOC calibration). The average response quantitation references are listed in Table 8 (multi-point calibration) and Table 8A (Tox/LOC calibration).

1. Calibration acceptance criteria

a. Ion abundance ratios must be within ion ratio limits described in Table 5. The ion ratio limits are derived from EPA 1668C, Table 8.

b. Native and labeled CBs signal-to-noise ratio must be > 10.

c. Relative retention times of all native and labeled CB's must be within the limits listed in Table 1.

d. The %RSD for the response factors is <20% for the native compounds.

If any of these criteria cannot be met, corrective actions are required to bring system within compliance. Possible corrective actions include cleaning/replacing ion volume and/or ion source, cleaning injection port and replacing injection port liner, trimming and/or replacing analytical column, modifying GC program to improve chromatographic resolution.

2. Initial Calibration Verification (ICV)

Following calibration and prior to sample analysis a second source standard is analyzed to verify initial calibration. The ICV must meet continuing calibration limits listed in Table 6. The ICV standard includes the Tox/LOC compounds and is prepared from the PAR stock solution at the same concentration as an OPR. An OPR extract may be used to verify initial calibration as long as all recoveries satisfy the continuing calibration limits listed in Table 6.

E. Sample data acquisition and data review.

Preparation and cleanup procedures for water samples to be analyzed by high resolution mass spectrometry are outlined in *Analysis #10914*. Preparation and cleanup procedures for solid samples to be analyzed by high resolution mass spectrometry are outlined in *Analysis #11030*.

Prior to injection on the HRGC/HRMS instrument, the extract is reduced to an estimated 10 µl with purified nitrogen and 10 µl of Internal Standards Spiking Solution (see "Reagents and Standards" section) for a final volume of 20 µl.

When the preparation of the extracts is completed, the Xcalibur Sequence Setup application is used to create a sequence of automated injections of sample extracts and acquire the sample data.

After the data has been acquired, process the data according to *T-HRMS-WI9433*. Data reduction criteria are outlined in the QA/QC section of this document.

Target compound detections exceeding signal intensity of 5 E7 are outside of the linear range of the detector and results may be biased. Where target compounds have exceeded the linear range of the detector a five-fold dilution is performed by adding 80 ul of Nonane to the extract. Likewise, an aliquot of the extract may be spiked into a volume of Nonane to achieve a five-fold dilution (for example, 4 ul sample extract into 16 ul Nonane). If the concentration of target analytes greatly exceeds the linear range of the instrument the sample should be re-extracted with sample amount of 1g. A five-fold dilution may be performed on the re-extracted sample. Further dilution of extracts typically results in ion abundance ratio failures for the labeled compounds and is not recommended. The addition of labeled compound standard during the dilution process introduces significant bias in final results.

Once the data has been reviewed, it is uploaded and verified in the LIMS database. The data reviewer double checks the estimated detection limit (EDL) against the limit of quantitation (LOQ) to ensure that the EDL < LOQ. An automated process also exists that alerts the data reviewer if the EDL > LOQ. If alerted, the LOQ is raised to match the EDL prior to the release of sample results.

F. DFS system performance verification

1. Mass Resolution of 10000.

Prior to analysis of the calibration verification the Resolution Check Procedure is acquired to document that the system meets the >10000 mass resolution requirement across the range of PFK masses used during the analysis of samples. The mass resolution check is performed at the beginning and end of every 12 hours. Mass resolution is also evaluated on the lock mass of each MID during analysis.

The resolution check procedure documents lock mass resolution and cali mass resolution for each descriptor. The procedure also documents mass resolution for all PFK masses listed on the PFK.lua file that exist within the mass range between lock and cali masses for each mass descriptor. At a minimum, the mass resolution of three PFK masses is documented for each MID.

2. Verification of system performance. (Calibration Verification)

Following the mass resolution check the calibration verification standard is injected prior to sample analysis. The calibration verification must meet the following criteria:

a. The chromatographic criteria listed in Procedure C 1 must be met.

b. The ion ratio; signal-to-noise ratio; and relative retention time criteria listed in Procedure D must be met.

c. The recovery criteria listed in Table 6 must be met when using the multi-point calibration for all 209 congeners. The recovery criteria listed in Table 6A must be met when using the Tox/LOC multi-point calibration with single point RF from the daily calibration verification for remaining congeners.

Calculations

A. The response factors (RF) calculated by TargetQuan for the quantitation of PCB congeners using the following formula:

$$RF = (SUMA * SPAis) / (SUMAis * SPA)$$

Where:

SUMA = sum of area of Quan Mass + area of Ratio Mass 1

SPAis = specified amount of internal standard (ng/mL)

SUMAis = sum of area of Quan Mass + Ratio Mass 1 for of internal standard

SPA = specified amount of standard (ng/mL)

B. The concentration of the native PCB congeners in the sample are calculated using the TargetQuan software using the following equation:

$$Concentration = ((SUMA * SPAis)/(SUMAis * ARFA)) * hSV/hSWT * hDF$$

Where:

SUMA = sum of area of Quan Mass + area of Ratio Mass 1

SPAis = specified amount of internal standard (ng/mL)

SUMAis = sum of area of Quan Mass + Ratio Mass 1 for internal standard

ARFA = actual RF based on area

hSV = final sample volume (µl)

hSWT = initial sample volume for waters (in liters) or initial sample weight for solids (in grams)

hDF = secondary dilution factor of extract.

Statistical Information/Method Performance

The OPR should contain all compounds listed in the method. OPR, MS, labeled compound recoveries and RPD are compared to the limits stored on the LIMS. These limits are specified in the method. Historical data for MS/Ds, OPRs, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to *QA-SOP11892* for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

The following QA/QC procedures are used to assure that data of known quality are generated using this method:

1. MS Resolution: Mass calibration and tuning of the instrument are performed prior to the start of analysis.. A static resolving power check must be done at the beginning and end of each 12 hour shift.

2. 209 PCB VER: As described in the DFS System Performance Verification section, a calibration verification (VER) standard is analyzed every 12 hours. The VER standard must meet the following acceptance criteria:

   a. The ion ratio specifications as shown in Table 5 (based on EPA 1668C, Table 8)

   b. The acceptance criteria listed in Table 6. (based on EPA 1668A and EPA 1668C, Table 6).

   c. The signal-to-noise ratio must be at least 10.

   d. The GC column performance meets criteria listed in EPA 1668C, section 6.9.1.1.

3. OPR: With every sample preparation batch (maximum 20 field samples), an ongoing precision and recovery sample (OPR) must be prepared and analyzed. The OPR must be analyzed prior to the samples of the associated batch. The samples and OPR QC sample must meet the acceptance criteria listed in EPA 1668A or EPA 1668C, Table 6, depending on project requirements. Client specified QC will supersede acceptance criteria listed in this SOP. See Table 6.

4. Method Blank: With every sample preparation batch (maximum 20 field samples), a method blank is required. The method blank must be run after the OPR to demonstrate freedom from contamination from the OPR analysis. The acceptable maximum levels to report in the method blank can be prescribed by specific project requirements. If no specific requirements exist, the EPA 1668C requirements will be followed which states that analytes cannot be detected at a concentration greater than 2X the Minimum Level. The maximum levels for any target PCB congeners or interference is listed in Table 4.

Samples associated with a non-compliant method blank or OPR are re-extracted. However, the sample data may be reported with a non-compliant method blank if the sample concentration is >10 times that of the method blank or if the compound present in the method blank is not detected in the sample. Samples with a non-compliant OPR may be reported if the OPR recovery is indicative of increased sensitivity and the compound is not detected in the sample.

5. Identification Criteria for Labeled and Non-Labeled Congeners:

For an analysis to be considered acceptable for reporting purposes, the following criteria must be met:

a. The quant mass and ratio mass of any given compound must maximize within ±2 scans of each other.

b. The ion ratio of the ratio mass to quant mass must fall within the limits listed in Table 5.

c. The relative retention time of the CB peak must be within the RRT Window criteria listed in Table 1.

d. The signal-to-noise ratio must be ≥10 for all CB's in the calibration and VER standards, and ≥2.5 for each CB detected in a sample.

e. The % Recovery of labeled and non-labeled congeners must meet the recovery limits listed in Table 6.

f. The lock mass for any reported congener level must not vary by more than +20% throughout the retention time window of the MID trace.

6. Interferences from high concentrations of fragments from loss of one or two chlorines from higher chlorinated closely eluting congeners may pose a problem in the determination of congeners 81, 123, 126, and 169. When high concentrations of closely eluting congeners are observed the lower chlorination congener result may be inflated or detected as a false positive. When interference is suspected the extract may be submitted to additional clean-up procedures.

Table 1

209 PCB Congener Retention Times

Compound Name	Entry Identifier	Quant Mass	Ratio Mass	Specified RT (min)	RRT Window (sec's)
PCB1	N1PCB1	190.0363 +/- 5 ppm	188.0393 +/- 5 ppm	10.27	-1 + 3
PCB2	N1PCB2	190.0363 +/- 5 ppm	188.0393 +/- 5 ppm	12.35	6
PCB3	N1PCB3	190.0363 +/- 5 ppm	188.0393 +/- 5 ppm	12.52	-1 + 3
PCB4	N2PCB4	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	12.78	-1 + 3
PCB10	N2PCB10	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	12.95	6
PCB9	N2PCB9	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	14.79	6
PCB7	N2PCB7	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	14.95	6
PCB6	N2PCB6	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	15.19	6
PCB5	N2PCB5	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	15.49	6
PCB8	N2PCB8	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	15.61	6
PCB19	N3PCB19	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	15.91	-1 + 3
PCB14	N2PCB14	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	17.22	6
PCB18+30	N3PCB18+30	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	17.72	6
PCB11	N2PCB11	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	18.06	6
PCB17	N3PCB17	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	18.15	6
PCB12+13	N2PCB12+13	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	18.36	6
PCB27	N3PCB27	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	18.37	6
PCB24	N3PCB24	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	18.49	6
PCB16	N3PCB16	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	18.61	6
PCB15	N2PCB15	223.9974 +/- 5 ppm	222.0003 +/- 5 ppm	18.66	-1 + 3
PCB54	N4PCB54	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	18.94	-1 + 3

PCB32	N3PCB32	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	19.11	6
PCB34	N3PCB34	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	20.34	6
PCB23	N3PCB23	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	20.49	6
PCB26+29	N3PCB26+29	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	20.8	10
PCB25	N3PCB25	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	21.01	6
PCB50+53	N4PCB50+53	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	21.05	10
PCB31	N3PCB31	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	21.32	6
PCB20+28	N3PCB20+28	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	21.62	10
PCB45	N4PCB45	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	21.68	10
PCB51	N4PCB51	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	21.75	10
PCB21+33	N3PCB21+33	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	21.82	10
PCB46	N4PCB46	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	21.97	6
PCB22	N3PCB22	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	22.21	6
PCB52	N4PCB52	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	23.35	6
PCB73	N4PCB73	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	23.48	6
PCB43	N4PCB43	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	23.56	6
PCB36	N3PCB36	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	23.7	6
PCB49+69	N4PCB49+69	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	23.78	10

Table 1 Continued – 209 PCB Congener Retention Times

Compound Name	Entry Identifier	Quant Mass	Ratio Mass	Specified RT (min)	RRT Window (sec's)
PCB39	N3PCB39	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	24.05	6
PCB48	N4PCB48	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	24.07	6
PCB104	N5PCB104	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	24.24	-1 + 3
PCB44+47+65	N4PCB44+47+65	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	24.3	10
PCB38	N3PCB38	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	24.59	6
PCB59+62+75	N4PCB59+62+75	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	24.61	10
PCB96	N5PCB96	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	24.59	10
PCB42	N4PCB42	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	24.79	6
PCB35	N3PCB35	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	25.04	6
PCB41	N4PCB41	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	25.14	10
PCB40+71	N4PCB40+71	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	25.26	10
PCB37	N3PCB37	257.9584 +/- 5 ppm	255.9613 +/- 5 ppm	25.43	-1 + 3
PCB64	N4PCB64	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	25.48	6
PCB72	N4PCB72	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	26.27	6
PCB103	N5PCB103	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	26.44	6
PCB68	N4PCB68	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	26.54	6

PCB94	N5PCB94	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	26.64	6
PCB57	N4PCB57	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	26.93	6
PCB95	N5PCB95	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	27.05	10
PCB93+100	N5PCB93+100	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	27.26	15
PCB58	N4PCB58	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	27.17	6
PCB98+102	N5PCB98+102	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	27.41	10
PCB67	N4PCB67	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	27.33	6
PCB88	N5PCB88	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	27.77	12
PCB63	N4PCB63	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	27.57	6
PCB91	N5PCB91	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	27.88	10
PCB61+70+74+76	N4PCB61+70+74+76	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	27.89	12
PCB84	N5PCB84	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	28.08	6
PCB66	N4PCB66	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	28.2	6
PCB55	N4PCB55	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	28.36	6
PCB89	N5PCB89	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	28.53	6
PCB56	N4PCB56	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	28.84	6
PCB121	N5PCB121	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	28.94	6
PCB60	N4PCB60	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	29.06	6
PCB92	N5PCB92	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	29.33	6
PCB80	N4PCB80	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	29.47	6
PCB155	N6PCB155	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	29.66	-1 + 3
PCB152	N6PCB152	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	29.88	6
PCB90+101+113	N5PCB90+101+113	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	29.91	10

Table 1 Continued – 209 PCB Congener Retention Times

Compound Name	Entry Identifier	Quant Mass	Ratio Mass	Specified RT (min)	RRT Window (sec's)
PCB150	N6PCB150	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	30.07	6
PCB83	N5PCB83	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	30.45	12
PCB136	N6PCB136	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	30.46	6
PCB99	N5PCB99	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	30.57	10
PCB112	N5PCB112	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	30.7	6
PCB145	N6PCB145	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	30.79	6
PCB86+87+97+109 +119+125	N5PCB86+87+97+ 109+119+125	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	31.13	16
PCB79	N4PCB79	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	31.15	6
PCB78	N4PCB78	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	31.82	6
PCB85+116+117	N5PCB85+116+117	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	32.01	12
PCB110+115	N5PCB110+115	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	32.26	10

PCB81	N4DLPCB81	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	32.38	-1 + 3
PCB82	N5PCB82	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	32.62	6
PCB148	N6PCB148	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	32.66	6
PCB77	N4DLPCB77	291.9194 +/- 5 ppm	289.9224 +/- 5 ppm	33.12	-1 + 3
PCB111	N5PCB111	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	33.17	6
PCB135+151	N6PCB135+151	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	33.44	10
PCB120	N5PCB120	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	33.8	6
PCB154	N6PCB154	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	33.8	10
PCB144	N6PCB144	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	34.23	6
PCB147+149	N6PCB147+149	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	34.78	10
PCB134	N6PCB134	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	35.07	10
PCB143	N6PCB143	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	35.22	10
PCB108+124	N5PCB108+124	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	35.49	10
PCB139+140	N6PCB139+140	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	35.67	10
PCB107	N5PCB107	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	35.87	6
PCB131	N6PCB131	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	35.99	6
PCB123	N5DLPCB123	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	36.06	-1 + 3
PCB106	N5PCB106	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	36.22	6
PCB142	N6PCB142	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	36.22	6
PCB118	N5DLPCB118	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	36.58	-1 + 3
PCB132	N6PCB132	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	36.74	10
PCB122	N5PCB122	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	37.11	6
PCB188	N7PCB188	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	37.4	-1 + 3
PCB114	N5DLPCB114	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	37.4	-1 + 3
PCB133	N6PCB133	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	37.58	6
PCB179	N7PCB179	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	37.92	6
PCB165	N6PCB165	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	38.2	6
PCB105	N5DLPCB105	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	38.41	-2 + 3

Table 1 Continued – 209 PCB Congener Retention Times

Compound Name	Entry Identifier	Quant Mass	Ratio Mass	Specified RT (min)	RRT Window (sec's)
PCB146	N6PCB146	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	38.56	6
PCB184	N7PCB184	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	38.72	6
PCB161	N6PCB161	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	38.76	6
PCB176	N7PCB176	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	39.23	6
PCB153+168	N6PCB153+168	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	39.51	10
PCB141	N6PCB141	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	39.75	6

PCB186	N7PCB186	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	39.88	6
PCB130	N6PCB130	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	40.34	6
PCB127	N5PCB127	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	40.59	6
PCB137	N6PCB137	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	40.64	6
PCB164	N6PCB164	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	40.82	6
PCB129+138+163	N6PCB129+138+163	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	41.26	14
PCB160	N6PCB160	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	41.43	10
PCB158	N6PCB158	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	41.76	6
PCB178	N7PCB178	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	41.88	6
PCB175	N7PCB175	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	42.72	6
PCB126	N5DLPCB126	327.8775 +/- 5 ppm	325.8804 +/- 5 ppm	42.79	-1 + 3
PCB128+166	N6PCB128+166	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	42.89	10
PCB187	N7PCB187	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	43.09	6
PCB182	N7PCB182	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	43.34	6
PCB183+185	N7PCB183+185	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	43.89	6
PCB174	N7PCB174	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	44.18	6
PCB159	N6PCB159	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	44.25	6
PCB162	N6PCB162	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	44.61	6
PCB177	N7PCB177	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	44.73	6
PCB202	N8PCB202	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	44.9	-1 + 3
PCB167	N6DLPCB167	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	45.21	-1 + 3
PCB181	N7PCB181	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	45.22	6
PCB171+173	N7PCB171+173	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	45.48	6
PCB201	N8PCB201	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	46.03	10
PCB156+157	N6DLPCB156+157	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	46.61	10
PCB204	N8PCB204	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	46.85	6
PCB197+200	N8PCB197+200	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	47.25	6
PCB172	N7PCB172	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	47.52	6
PCB192	N7PCB192	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	47.85	6
PCB180+193	N7PCB180+193	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	48.24	6
PCB191	N7PCB191	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	48.69	6
PCB170	N7PCB170	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	49.71	6
PCB190	N7PCB190	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	50.31	6

Table 1 Continued – 209 PCB Congener Retention Times

Compound Name	Entry Identifier	Quant Mass	Ratio Mass	Specified RT (min)	RRT Window (sec's)
PCB169	N6DLPCB169	361.8385 +/- 5 ppm	359.8415 +/- 5 ppm	50.4	-1 + 3

PCB198+199	N8PCB198+199	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	50.5	10
PCB196	N8PCB196	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	51.26	6
PCB203	N8PCB203	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	51.46	6
PCB208	N9PCB208	463.7216 +/- 5 ppm	461.7246 +/- 5 ppm	52.67	-1 + 3
PCB195	N8PCB195	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	52.9	6
PCB189	N7DLPCB189	395.7995 +/- 5 ppm	393.8025 +/- 5 ppm	53.19	-1 + 3
PCB207	N9PCB207	463.7216 +/- 5 ppm	461.7246 +/- 5 ppm	53.66	6
PCB194	N8PCB194	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	55.42	6
PCB205	N8PCB205	429.7606 +/- 5 ppm	427.7635 +/- 5 ppm	55.9	-1 + 3
PCB206	N9PCB206	463.7216 +/- 5 ppm	461.7246 +/- 5 ppm	57.75	-1 + 3
PCB209	N10PCB209	499.6797 +/- 5 ppm	497.6826 +/- 5 ppm	59.64	-1 + 3
13C12-PCB28	C3PCB28	269.9986 +/- 5 ppm	268.0016 +/- 5 ppm	21.57	20
13C12-PCB111	C5PCB111	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	33.14	20
13C12-PCB178	C7PCB178	407.8398 +/- 5 ppm	405.8428 +/- 5 ppm	41.85	20
13C12-PCB9	I2PCB9	236.0376 +/- 5 ppm	234.0406 +/- 5 ppm	14.77	25
13C12-PCB52	I4PCB52	303.9597 +/- 5 ppm	301.9626 +/- 5 ppm	23.33	25
13C12-PCB101	I5PCB101	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	29.91	25
13C12-PCB138	I6PCB138	373.8788 +/- 5 ppm	371.8817 +/- 5 ppm	41.19	100
13C12-PCB194	I8PCB194	441.8008 +/- 5 ppm	439.8038 +/- 5 ppm	55.4	25
13C12-PCB1	L1PCB1	202.0766 +/- 5 ppm	200.0795 +/- 5 ppm	10.25	30
13C12-PCB3	L1PCB3	202.0766 +/- 5 ppm	200.0795 +/- 5 ppm	12.51	30
13C12-PCB4	L2PCB4	236.0376 +/- 5 ppm	234.0406 +/- 5 ppm	12.75	30
13C12-PCB8	LA2PCB8	236.0376 +/- 5 ppm	234.0406 +/- 5 ppm	15.6	20
13C12-PCB19	L3PCB19	269.9986 +/- 5 ppm	268.0016 +/- 5 ppm	15.89	30
13C12-PCB15	L2PCB15	236.0376 +/- 5 ppm	234.0406 +/- 5 ppm	18.65	20
13C12-PCB54	L4PCB54	303.9597 +/- 5 ppm	301.9626 +/- 5 ppm	18.9	20
13C12-PCB32	LA3PCB32	269.9986 +/- 5 ppm	268.0016 +/- 5 ppm	19.09	30
13C12-PCB31	LA3PCB31	269.9986 +/- 5 ppm	268.0016 +/- 5 ppm	21.3	30
13C12-PCB104	L5PCB104	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	24.2	20
13C12-PCB47	LA4PCB47	303.9597 +/- 5 ppm	301.9626 +/- 5 ppm	24.3	20
13C12-PCB37	L3PCB37	269.9986 +/- 5 ppm	268.0016 +/- 5 ppm	25.41	30
13C12-PCB95	LA5PCB95	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	27.04	20
13C12-PCB70	LA4PCB70	303.9597 +/- 5 ppm	301.9626 +/- 5 ppm	27.86	20
13C12-PCB60	LA4PCB60	303.9597 +/- 5 ppm	301.9626 +/- 5 ppm	29.02	20
13C12-PCB155	L6PCB155	373.8788 +/- 5 ppm	371.8817 +/- 5 ppm	29.64	20
13C12-PCB85	LA5PCB85	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	31.99	20
13C12-PCB81	L4PCB81	303.9597 +/- 5 ppm	301.9626 +/- 5 ppm	32.35	20
13C12-PCB77	L4PCB77	303.9597 +/- 5 ppm	301.9626 +/- 5 ppm	33.09	20

Table 1 Continued – 209 PCB Congener Retention Times

Compound Name	Entry Identifier	Quant Mass	Ratio Mass	Specified RT (min)	RRT Window (sec's)
13C12-PCB123	L5PCB123	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	36.02	20
13C12-PCB118	L5PCB118	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	36.54	20
13C12-PCB188	L7PCB188	407.8398 +/- 5 ppm	405.8428 +/- 5 ppm	37.37	20
13C12-PCB114	L5PCB114	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	37.37	20
13C12-PCB133	LA6PCB133	373.8788 +/- 5 ppm	371.8817 +/- 5 ppm	37.53	20
13C12-PCB105	L5PCB105	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	38.37	20
13C12-PCB141	LA6PCB141	373.8788 +/- 5 ppm	371.8817 +/- 5 ppm	39.72	20
13C12-PCB127	LA5PCB127	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	40.57	20
13C12-PCB126	L5PCB126	339.9178 +/- 5 ppm	337.9207 +/- 5 ppm	42.75	20
13C12-PCB128	LA6PCB128	373.8788 +/- 5 ppm	371.8817 +/- 5 ppm	42.89	20
13C12-PCB162	LA6PCB162	373.8788 +/- 5 ppm	371.8817 +/- 5 ppm	44.57	20
13C12-PCB202	L8PCB202	441.8008 +/- 5 ppm	439.8038 +/- 5 ppm	44.86	20
13C12-PCB167	L6PCB167	373.8788 +/- 5 ppm	371.8817 +/- 5 ppm	45.17	20
13C12-PCB156+157	L6PCB156+157	373.8788 +/- 5 ppm	371.8817 +/- 5 ppm	46.58	20
13C12-PCB180	LA7PCB180	407.8398 +/- 5 ppm	405.8428 +/- 5 ppm	48.26	20
13C12-PCB169	L6PCB169	373.8788 +/- 5 ppm	371.8817 +/- 5 ppm	50.38	20
13C12-PCB208	L9PCB208	475.7619 +/- 5 ppm	473.7648 +/- 5 ppm	52.63	20
13C12-PCB189	L7PCB189	407.8398 +/- 5 ppm	405.8428 +/- 5 ppm	53.15	20
13C12-PCB205	L8PCB205	441.8008 +/- 5 ppm	439.8038 +/- 5 ppm	55.89	30
13C12-PCB206	L9PCB206	475.7619 +/- 5 ppm	473.7648 +/- 5 ppm	57.73	30
13C12-PCB209	L10PCB209	511.7199 +/- 5 ppm	509.7229 +/- 5 ppm	59.6	30

Table 2

Typical Retention Times for MID switches

MID	Start	Measure	End	Cycletime	First PCB	Last PCB
# 1	10:00 min	4:11 min	14:11 min	1.00 sec		PCB10
# 2	14:11 min	6:11 min	20:23 min	1.00 sec	PCB9	PCB32
# 3	20:23 min	6:36 min	27:00 min	1.00 sec	PCB34	PCB64
# 4	27:00 min	7:42 min	34:42 min	1.00 sec	PCB72	PCB144
# 5	34:42 min	6:11 min	40:53 min	1.00 sec	PCB147+149	PCB182
# 6	40:53 min	7:18 min	48:12 min	1.00 sec	PCB183	PCB203

# 7	48:12 min	6:00 min	54:12 min	1.00 sec	PCB208	PCB206
# 8	54:12 min	3:37 min	58:00 min	1.00 sec	PCB209	

Table 3

PCB Congener MID

Window # 1				
mass	F	int	gr	time (ms)
180.9883	I	20	1	7
188.0388		1	1	151
190.0358		1	1	151
200.0790		5	1	30
202.0761		5	1	30
221.9998		1	1	151
223.9969		1	1	151
225.9939		1	1	151
234.0401		5	1	30
236.0371		5	1	30
242.9851	c	20	1	7
Window # 2				
mass	F	int	gr	time (ms)
218.9851	I	20	1	5
221.9998		1	1	119
223.9969		1	1	119
234.0401		5	1	23
236.0371		5	1	23
255.9608		1	1	119
257.9579		1	1	119
268.0011		5	1	23
269.9981		5	1	23
280.9819	c	20	1	5
289.9219		1	1	119
291.9189		1	1	119
301.9621		5	1	23
303.9592		5	1	23

Window # 3				
mass	F	int	gr	time (ms)
255.9608		1	1	119
257.9579		1	1	119
268.0011		5	1	23
269.9981		5	1	23
280.9819	1	20	1	5
289.9219		1	1	119
291.9189		1	1	119
301.9621		5	1	23
303.9592		5	1	23
325.8799		1	1	119
327.877		1	1	119
330.9787	c	20	1	5
337.9202		5	1	23
339.9173		5	1	23
Window # 4				
mass	F	int	gr	time (ms)
280.9819	I	20	1	5
289.9219		1	1	119
291.9189		1	1	119
301.9621		5	1	23
303.9592		5	1	23
325.8799		1	1	119
327.8770		1	1	119
330.9787	c	20	1	5
337.9202		5	1	23
339.9173		5	1	23
359.8410		1	1	119
361.8380		1	1	119
371.8812		5	1	23
373.8783		5	1	23

Table 3 Continued – PCB Congener MID

Window # 5				
mass	F	int	gr	time (ms)

Window # 7				
mass	F	int	gr	time (ms)

325.8799		1	1	119
327.8770		1	1	119
330.9787	I	20	1	5
337.9202		5	1	23
339.9173		5	1	23
359.8410		1	1	119
361.8380		1	1	119
371.8812		5	1	23
373.8783		5	1	23
393.8020		1	1	119
395.7990		1	1	119
404.9755	c	20	1	5
405.8423		5	1	23
407.8393		5	1	23
Window # 6				
mass	F	int	gr	time (ms)
330.9787	I	20	1	5
359.8410		1	1	119
361.8380		1	1	119
371.8812		5	1	23
373.8783		5	1	23
393.8020		1	1	119
395.7990		1	1	119
404.9795	c	20	1	5
405.8423		5	1	23
407.8393		5	1	23
427.7630		1	1	119
429.7601		1	1	119
439.8033		5	1	23
441.8003		5	1	23

393.8020		1	1	119
395.7990		1	1	119
404.9755	I	20	1	5
405.8423		5	1	23
407.8393		5	1	23
427.7630		1	1	119
429.7601		1	1	119
439.8033		5	1	23
441.8003		5	1	23
454.9723	c	20	1	5
461.7241		1	1	119
463.7211		1	1	119
473.7643		5	1	23
475.7614		5	1	23
Window # 8				
mass	F	int	gr	time (ms)
454.9723	I	20	1	18
492.9691	c	20	1	18
497.6821		1	1	364
499.6792		1	1	364
509.7224		5	1	72
511.7194		5	1	72

Table 4

Maximum Levels in DL-PCB Method Blank

Compound Name	Water Blank Limit (pg/l)	Solid Blank Limit (ng/kg)
PCB1	40	4
PCB3	100	10
PCB4	100	10

PCB19	40	4
PCB15	100	10
PCB54	100	10
PCB104	100	10
PCB37	40	4
PCB155	100	10
PCB81	100	10
PCB77	100	10
PCB123	100	10
PCB118	200	20
PCB114	100	10
PCB188	100	10
PCB105	100	10
PCB126	100	10
PCB202	200	20
PCB167	100	10
PCB156-157	200	20
PCB169	100	10
PCB208	100	10
PCB189	100	10
PCB205	100	10
PCB206	100	10
PCB209	100	10

Table 5

PCB Congener Ion Ratio Limits

Compound Name	Ion Ratio Limit	Compound Name	Ion Ratio Limit
PCB1	2.6550 - 3.6050	13C12-PCB1	2.6550 - 3.6050
PCB3	2.6550 - 3.6050	13C12-PCB3	2.6550 - 3.6050
PCB4	1.3250 - 1.7950	13C12-PCB4	1.3250 - 1.7950
PCB19	0.8750 - 1.2050	13C12-PCB8	1.3250 - 1.7950
PCB15	1.3250 - 1.7950	13C12-PCB19	0.8750 - 1.2050
PCB54	0.6450 - 0.8950	13C12-PCB15	1.3250 - 1.7950
PCB104	1.3150 - 1.7850	13C12-PCB54	0.6450 - 0.8950
PCB37	0.8750 - 1.2050	13C12-PCB32	0.8750 - 1.2050
PCB155	1.0450 - 1.4350	13C12-PCB31	0.8750 - 1.2050
PCB81	0.6450 - 0.8950	13C12-PCB104	1.3150 - 1.7850
PCB77	0.6450 - 0.8950	13C12-PCB47	0.6450 - 0.8950
PCB123	1.3150 - 1.7850	13C12-PCB37	0.8750 - 1.2050
PCB118	1.3150 - 1.7850	13C12-PCB95	1.3150 - 1.7850
PCB114	1.3150 - 1.7850	13C12-PCB70	0.6450 - 0.8950
PCB188	0.8850 - 1.2150	13C12-PCB60	0.6450 - 0.8950
PCB105	1.3150 - 1.7850	13C12-PCB155	1.0450 - 1.4350

PCB126	1.3150 -	1.7850
PCB202	0.7550 -	1.0250
PCB167	1.0450 -	1.4350
PCB156-157	1.0450 -	1.4350
PCB169	1.0450 -	1.4350
PCB208	0.6450 -	0.8950
PCB189	0.8850 -	1.2150
PCB205	0.7550 -	1.0250
PCB206	0.6450 -	0.8950
PCB209	0.9850 -	1.3350
Other mono-Cl PCBs	2.6550 -	3.6050
Other di-Cl PCBs	1.3250 -	1.7950
Other tri-Cl PCBs	0.8750 -	1.2050
Other tetra-Cl PCBs	0.6450 -	0.8950
Other penta-Cl PCBs	1.3150 -	1.7850
Other hexa-Cl PCBs	1.0450 -	1.4350
Other hepta-Cl PCBs	0.8850 -	1.2150
Other octa-Cl PCBs	0.7550 -	1.0250
Other nona-Cl PCBs	0.6450 -	0.8950
13C12-PCB9	1.3250 -	1.7949
13C12-PCB52	0.6450 -	0.8950
13C12-PCB101	1.3150 -	1.7850
13C12-PCB138	1.0450 -	1.4350
13C12-PCB194	0.7550 -	1.0250
13C12-PCB28	0.8750 -	1.2050
13C12-PCB111	1.3150 -	1.7850
13C12-PCB178	0.8850 -	1.2150

13C12-PCB85	1.3150 -	1.7850
13C12-PCB81	0.6450 -	0.8950
13C12-PCB77	0.6450 -	0.8950
13C12-PCB123	1.3150 -	1.7850
13C12-PCB118	1.3150 -	1.7850
13C12-PCB188	0.8850 -	1.2150
13C12-PCB114	1.3150 -	1.7850
13C12-PCB133	1.0450 -	1.4350
13C12-PCB105	1.3150 -	1.7850
13C12-PCB141	1.0450 -	1.4350
13C12-PCB127	1.3150 -	1.7850
13C12-PCB126	1.3150 -	1.7850
13C12-PCB128	1.0450 -	1.4350
13C12-PCB162	1.0450 -	1.4350
13C12-PCB202	0.7550 -	1.0250
13C12-PCB167	1.0450 -	1.4350
13C12-PCB156+157	1.0450 -	1.4350
13C12-PCB180	0.8850 -	1.2150
13C12-PCB169	1.0450 -	1.4350
13C12-PCB208	0.6450 -	0.8950
13C12-PCB189	0.8850 -	1.2150
13C12-PCB205	0.7550 -	1.0250
13C12-PCB206	0.6450 -	0.8950
13C12-PCB209	0.9850 -	1.3350

Table 6

PCB Congener QC Acceptance Criteria

Compound Name	VER Specified Amt (ng/ml)	1668A VER %REC limits	1668C VER %REC limits	OPR Specified Amt in extract (ng/ml)	1668A OPR %REC Limits	1668C OPR %REC Limits
PCB1	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB2	25	70 - 130	75 - 125		-	
PCB3	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB4	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB10	25	70 - 130	75 - 125		-	
PCB9	25	70 - 130	75 - 125		-	
PCB7	25	70 - 130	75 - 125		-	

PCB6	25	70 - 130	75 - 125		-	
PCB5	25	70 - 130	75 - 125		-	
PCB8	25	70 - 130	75 - 125		-	
PCB19	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB14	25	70 - 130	75 - 125		-	
PCB18+30	50	70 - 130	75 - 125		-	
PCB11	25	70 - 130	75 - 125		-	
PCB17	25	70 - 130	75 - 125		-	
PCB12+13	50	70 - 130	75 - 125		-	
PCB27	25	70 - 130	75 - 125		-	
PCB24	25	70 - 130	75 - 125		-	
PCB16	25	70 - 130	75 - 125		-	
PCB15	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB54	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB32	25	70 - 130	75 - 125		-	
PCB34	25	70 - 130	75 - 125		-	
PCB23	25	70 - 130	75 - 125		-	
PCB26+29	50	70 - 130	75 - 125		-	
PCB25	25	70 - 130	75 - 125		-	
PCB50+53	100	70 - 130	75 - 125		-	
PCB31	25	70 - 130	75 - 125		-	
PCB20+28	50	70 - 130	75 - 125		-	
PCB45	50	70 - 130	75 - 125		-	
PCB51	50	70 - 130	75 - 125		-	
PCB21+33	50	70 - 130	75 - 125		-	
PCB46	50	70 - 130	75 - 125		-	
PCB22	25	70 - 130	75 - 125		-	
PCB52	50	70 - 130	75 - 125		-	
PCB73	50	70 - 130	75 - 125		-	
PCB43	50	70 - 130	75 - 125		-	
PCB36	25	70 - 130	75 - 125		-	
PCB49+69	100	70 - 130	75 - 125		-	
PCB39	25	70 - 130	75 - 125		-	

Table 6 continued – PCB Congener QC Acceptance Criteria

Compound Name		1668A	1668C		1668A	1668C

	VER Specified Amt (ng/ml)	VER %REC limits	VER %REC limits	OPR Specified Amt in extract (ng/ml)	OPR %REC Limits	OPR %REC Limits
PCB48	50	70 - 130	75 - 125			
PCB104	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB44+47+65	150	70 - 130	75 - 125	-	-	-
PCB38	25	70 - 130	75 - 125	-	-	-
PCB59+62+75	150	70 - 130	75 - 125		-	
PCB96	50	70 - 130	75 - 125		-	
PCB42	50	70 - 130	75 - 125		-	
PCB35	25	70 - 130	75 - 125		-	
PCB41	50	70 - 130	75 - 125		-	
PCB40+71	100	70 - 130	75 - 125		-	
PCB37	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB64	50	70 - 130	75 - 125		-	
PCB72	50	70 - 130	75 - 125		-	
PCB103	50	70 - 130	75 - 125		-	
PCB68	50	70 - 130	75 - 125		-	
PCB94	50	70 - 130	75 - 125		-	
PCB57	50	70 - 130	75 - 125		-	
PCB95	50	70 - 130	75 - 125		-	
PCB93+100	100	70 - 130	75 - 125		-	
PCB58	50	70 - 130	75 - 125	-	-	-
PCB98+102	100	70 - 130	75 - 125	-	-	-
PCB67	50	70 - 130	75 - 125		-	
PCB88	50	70 - 130	75 - 125		-	
PCB63	50	70 - 130	75 - 125		-	
PCB91	50	70 - 130	75 - 125		-	
PCB61+70+74+76	200	70 - 130	75 - 125		-	
PCB84	50	70 - 130	75 - 125		-	
PCB66	50	70 - 130	75 - 125		-	
PCB55	50	70 - 130	75 - 125		-	
PCB89	50	70 - 130	75 - 125		-	
PCB56	50	70 - 130	75 - 125		-	
PCB121	50	70 - 130	75 - 125		-	
PCB60	50	70 - 130	75 - 125		-	
PCB92	50	70 - 130	75 - 125		-	
PCB80	50	70 - 130	75 - 125		-	
PCB155	50	70 - 130	75 - 125	50	50 - 150	60 - 135

PCB152	50	70 - 130	75 - 125		-	
PCB90+101+113	150	70 - 130	75 - 125		-	
PCB150	50	70 - 130	75 - 125		-	
PCB83	50	70 - 130	75 - 125		-	

Table 6 continued – PCB Congener QC Acceptance Criteria

Compound Name	VER Specified Amt (ng/ml)	1668A VER %REC limits	1668C VER %REC limits	OPR Specified Amt in extract (ng/ml)	1668A OPR %REC Limits	1668C OPR %REC Limits
PCB136	50	70 - 130	75 - 125	-	-	-
PCB99	50	70 - 130	75 - 125		-	
PCB112	50	70 - 130	75 - 125	-	-	-
PCB145	50	70 - 130	75 - 125	-	-	-
PCB86+87+97+109+119+125	300	70 - 130	75 - 125		-	
PCB79	50	70 - 130	75 - 125		-	
PCB78	50	70 - 130	75 - 125		-	
PCB85+116+117	150	70 - 130	75 - 125		-	
PCB110+115	100	70 - 130	75 - 125		-	
PCB81	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB82	50	70 - 130	75 - 125	-	-	-
PCB148	50	70 - 130	75 - 125		-	
PCB77	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB111	50	70 - 130	75 - 125		-	
PCB135+151	100	70 - 130	75 - 125		-	
PCB120	50	70 - 130	75 - 125		-	
PCB154	50	70 - 130	75 - 125		-	
PCB144	50	70 - 130	75 - 125		-	
PCB147+149	100	70 - 130	75 - 125		-	
PCB134	50	70 - 130	75 - 125	-	-	-
PCB143	50	70 - 130	75 - 125	-	-	-
PCB108+124	100	70 - 130	75 - 125		-	
PCB139+140	100	70 - 130	75 - 125		-	
PCB107	50	70 - 130	75 - 125		-	
PCB131	50	70 - 130	75 - 125		-	
PCB123	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB106	50	70 - 130	75 - 125		-	
		70 - 130			-	

PCB142	50		75 - 125			
PCB118	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB132	50	70 - 130	75 - 125		-	
PCB122	50	70 - 130	75 - 125		-	
PCB188	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB114	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB133	50	70 - 130	75 - 125		-	
PCB179	50	70 - 130	75 - 125		-	
PCB165	50	70 - 130	75 - 125		-	
PCB105	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB146	50	70 - 130	75 - 125		-	
PCB184	50	70 - 130	75 - 125		-	
PCB161	50	70 - 130	75 - 125		-	

Table 6 continued – PCB Congener QC Acceptance Criteria

Compound Name	VER Specified Amt (ng/ml)	1668A VER %REC limits	1668C VER %REC limits	OPR Specified Amt in extract (ng/ml)	1668A OPR %REC Limits	1668C OPR %REC Limits
PCB176	50	70 - 130	75 - 125	-	-	-
PCB153+168	100	70 - 130	75 - 125		-	
PCB141	50	70 - 130	75 - 125	-	-	-
PCB186	50	70 - 130	75 - 125	-	-	-
PCB130	50	70 - 130	75 - 125		-	
PCB127	50	70 - 130	75 - 125		-	
PCB137	50	70 - 130	75 - 125		-	
PCB164	50	70 - 130	75 - 125		-	
PCB129+138+163	150	70 - 130	75 - 125		-	
PCB160	50	70 - 130	75 - 125		-	
PCB158	50	70 - 130	75 - 125	-	-	-
PCB178	50	70 - 130	75 - 125		-	
PCB175	50	70 - 130	75 - 125		-	
PCB126	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB128+166	100	70 - 130	75 - 125		-	
PCB187	50	70 - 130	75 - 125		-	
PCB182	50	70 - 130	75 - 125		-	
PCB183+185	100	70 - 130	75 - 125		-	
PCB174	50	70 - 130	75 - 125		-	

PCB159	50	70 - 130	75 - 125	-	-	-
PCB162	50	70 - 130	75 - 125	-	-	-
PCB177	50	70 - 130	75 - 125		-	
PCB202	75	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB167	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB181	50	70 - 130	75 - 125		-	
PCB171+173	100	70 - 130	75 - 125		-	
PCB201	75	70 - 130	75 - 125		-	
PCB156+157	100	70 - 130	75 - 125	100	50 - 150	60 - 135
PCB204	75	70 - 130	75 - 125		-	
PCB197+200	150	70 - 130	75 - 125		-	
PCB172	50	70 - 130	75 - 125		-	
PCB192	50	70 - 130	75 - 125		-	
PCB180+193	100	70 - 130	75 - 125		-	
PCB191	50	70 - 130	75 - 125		-	
PCB170	50	70 - 130	75 - 125		-	
PCB190	50	70 - 130	75 - 125		-	
PCB169	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB198+199	150	70 - 130	75 - 125		-	
PCB196	75	70 - 130	75 - 125		-	
PCB203	75	70 - 130	75 - 125		-	

Table 6 continued – PCB Congener QC Acceptance Criteria

Compound Name	VER Specified Amt (ng/ml)	1668A VER %REC limits	1668C VER %REC limits	OPR Specified Amt in extract (ng/ml)	1668A OPR %REC Limits	1668C OPR %REC Limits
PCB208	75	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB195	75	70 - 130	75 - 125		-	
PCB189	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB207	75	70 - 130	75 - 125	-	-	-
PCB194	75	70 - 130	75 - 125		-	
PCB205	75	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB206	75	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB209	75	70 - 130	75 - 125	50	50 - 150	60 - 135
13C12-PCB28	100	60 - 130	75 - 125	50	40 - 125	15 - 145
13C12-PCB111	100	60 - 130	75 - 125	50	40 - 125	40 - 145
13C12-PCB178	100	60 - 130	75 - 125	50	40 - 125	40 - 145

13C12-PCB1	100	50 - 150	75 - 125	100	15 - 140	15 - 145
13C12-PCB1	100	50 - 150	75 - 125	100	15 - 140	15 - 145
13C12-PCB3	100	50 - 150	75 - 125	100	30 - 140	15 - 145
13C12-PCB4	100	50 - 150	75 - 125	100	30 - 140	15 - 145
13C12-PCB8	100	50 - 150	75 - 125	100	30 - 140	15 - 145
13C12-PCB19	100	50 - 150	75 - 125	100	30 - 140	15 - 145
13C12-PCB15	100	50 - 150	75 - 125	100	30 - 140	15 - 145
13C12-PCB54	100	50 - 150	75 - 125	100	30 - 140	15 - 145
13C12-PCB32	100	50 - 150	75 - 125	100	30 - 140	15 - 145
13C12-PCB31	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB104	100	50 - 150	75 - 125	100	30 - 140	15 - 145
13C12-PCB47	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB37	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB95	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB70	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB60	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB155	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB85	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB81	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB77	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB123	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB118	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB188	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB114	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB133	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB105	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB141	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB127	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB126	100	50 - 150	75 - 125	100	30 - 140	40 - 145

Table 6 continued – PCB Congener QC Acceptance Criteria

Compound Name	VER Specified Amt (ng/ml)	1668A VER %REC limits	1668C VER %REC limits	OPR Specified Amt in extract (ng/ml)	1668A OPR %REC Limits	1668C OPR %REC Limits
13C12-PCB128	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB162	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB202	100	50 - 150	75 - 125	100	30 - 140	40 - 145

13C12-PCB167	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB156+157	200	50 - 150	75 - 125	200	30 - 140	40 - 145
13C12-PCB180	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB169	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB208	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB189	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB205	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB206	100	50 - 150	75 - 125	100	30 - 140	40 - 145
13C12-PCB209	100	50 - 150	75 - 125	100	30 - 140	40 - 145

Table 6 continued – PCB Congener QC Acceptance Criteria

Compound Name	Sample Specified Amt (ng/ml)	1668A Sample %REC Limits	1668C Sample %REC Limits
13C12-PCB28	50	30 - 135	5 - 145
13C12-PCB111	50	30 - 135	10 - 145
13C12-PCB178	50	30 - 135	10 - 145
13C12-PCB1	100	15 - 150	5 - 145
13C12-PCB3	100	15 - 150	5 - 145
13C12-PCB4	100	25 - 150	5 - 145
13C12-PCB8	100	25 - 150	5 - 145
13C12-PCB19	100	25 - 150	5 - 145
13C12-PCB15	100	25 - 150	5 - 145
13C12-PCB54	100	25 - 150	5 - 145
13C12-PCB32	100	25 - 150	5 - 145
13C12-PCB31	100	25 - 150	5 - 145
13C12-PCB104	100	25 - 150	10 - 145
13C12-PCB47	100	25 - 150	5 - 145
13C12-PCB37	100	25 - 150	5 - 145
13C12-PCB95	100	25 - 150	10 - 145
13C12-PCB70	100	25 - 150	10 - 145
13C12-PCB60	100	25 - 150	10 - 145
13C12-PCB155	100	25 - 150	10 - 145
13C12-PCB85	100	25 - 150	10 - 145
13C12-PCB81	100	25 - 150	10 - 145
13C12-PCB77	100	25 - 150	10 - 145
13C12-PCB123	100	25 - 150	10 - 145
13C12-PCB118	100	25 - 150	10 - 145

13C12-PCB188	100	25 - 150	10 - 145
13C12-PCB114	100	25 - 150	10 - 145
13C12-PCB133	100	25 - 150	10 - 145
13C12-PCB105	100	25 - 150	10 - 145
13C12-PCB141	100	25 - 150	10 - 145
13C12-PCB127	100	25 - 150	10 - 145
13C12-PCB126	100	25 - 150	10 - 145
13C12-PCB128	100	25 - 150	10 - 145
13C12-PCB162	100	25 - 150	10 - 145
13C12-PCB202	100	25 - 150	10 - 145
13C12-PCB167	100	25 - 150	10 - 145
13C12-PCB156+157	100	25 - 150	10 - 145
13C12-PCB180	100	25 - 150	10 - 145
13C12-PCB169	100	25 - 150	10 - 145
13C12-PCB208	100	25 - 150	10 - 145
13C12-PCB189	100	25 - 150	10 - 145
13C12-PCB205	100	25 - 150	10 - 145
13C12-PCB206	100	25 - 150	10 - 145
13C12-PCB209	100	25 - 150	10 - 145

Table 6A

PCB Congener QC Acceptance Criteria Tox/LOC Calibration

Compound Name	VER Specified Amt (ng/ml)	1668A VER %REC limits	1668C VER %REC limits	OPR Specified Amt in extract (ng/ml)	1668A OPR %REC Limits	1668C OPR %REC Limits
PCB1	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB3	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB4	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB19	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB15	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB54	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB104	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB37	25	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB155	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB81	50	70 - 130	75 - 125	50	50 - 150	60 - 135

PCB77	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB123	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB118	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB188	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB114	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB105	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB126	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB202	75	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB167	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB156+157	100	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB169	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB208	75	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB189	50	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB205	75	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB206	75	70 - 130	75 - 125	50	50 - 150	60 - 135
PCB209	75	70 - 130	75 - 125	50	50 - 150	60 - 135
13C12-PCB28	100	60 - 130	65 - 135	50	40 - 125	15 - 145
13C12-PCB111	100	60 - 130	75 - 125	50	40 - 125	40 - 145
13C12-PCB178	100	60 - 130	75 - 125	50	40 - 125	40 - 145
13C12-PCB1	100	50 - 150	75 - 125	100	15 - 140	15 - 145
13C12-PCB3	100	50 - 150	50 - 145	100	15 - 140	15 - 145
13C12-PCB4	100	50 - 150	50 - 145	100	30 - 140	15 - 145
13C12-PCB19	100	50 - 150	50 - 145	100	30 - 140	15 - 145
13C12-PCB15	100	50 - 150	50 - 145	100	30 - 140	15 - 145
13C12-PCB54	100	50 - 150	50 - 145	100	30 - 140	15 - 145
13C12-PCB104	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB37	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB155	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB81	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB77	100	50 - 150	50 - 145	100	30 - 140	40 - 145

Table 6A continued – PCB Congener QC Acceptance Criteria Tox/LOC Calibration

Compound Name	VER Specified Amt (ng/ml)	1668A VER %REC limits	1668C VER %REC limits	OPR Specified Amt in extract (ng/ml)	1668A OPR %REC Limits	1668C OPR %REC Limits
13C12-PCB123	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB118	100	50 - 150	50 - 145	100	30 - 140	40 - 145

13C12-PCB188	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB114	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB105	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB126	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB202	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB167	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB156+157	200	50 - 150	50 - 145	200	30 - 140	40 - 145
13C12-PCB169	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB208	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB189	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB205	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB206	100	50 - 150	50 - 145	100	30 - 140	40 - 145
13C12-PCB209	100	50 - 150	50 - 145	100	30 - 140	40 - 145

Table 6A continued – PCB Congener QC Acceptance Criteria Tox/LOC Calibration

Compound Name	Sample Specified Amt (ng/ml)	1668A Sample %REC Limits	1668C Sample %REC Limits
13C12-PCB28	50	30 - 135	5 - 145
13C12-PCB111	50	30 - 135	10 - 145
13C12-PCB178	50	30 - 135	10 - 145
13C12-PCB1	100	15 - 150	5 - 145
13C12-PCB3	100	15 - 150	5 - 145
13C12-PCB4	100	25 - 150	5 - 145
13C12-PCB19	100	25 - 150	5 - 145
13C12-PCB15	100	25 - 150	5 - 145
13C12-PCB54	100	25 - 150	5 - 145
13C12-PCB104	100	25 - 150	10 - 145
13C12-PCB37	100	25 - 150	5 - 145
13C12-PCB155	100	25 - 150	10 - 145
13C12-PCB81	100	25 - 150	10 - 145
13C12-PCB77	100	25 - 150	10 - 145
13C12-PCB123	100	25 - 150	10 - 145
13C12-PCB118	100	25 - 150	10 - 145
13C12-PCB188	100	25 - 150	10 - 145
13C12-PCB114	100	25 - 150	10 - 145
13C12-PCB105	100	25 - 150	10 - 145

13C12-PCB126	100	25 - 150	10 - 145
13C12-PCB202	100	25 - 150	10 - 145
13C12-PCB167	100	25 - 150	10 - 145
13C12-PCB156+157	200	25 - 150	10 - 145
13C12-PCB169	100	25 - 150	10 - 145
13C12-PCB208	100	25 - 150	10 - 145
13C12-PCB189	100	25 - 150	10 - 145
13C12-PCB205	100	25 - 150	10 - 145
13C12-PCB206	100	25 - 150	10 - 145
13C12-PCB209	100	25 - 150	10 - 145

Table 7

PCB Congener Retention Time and Quantitation References

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
13C12-PCB9	I2PCB9		
13C12-PCB1	L1PCB1	I2PCB9	I2PCB9
PCB1	N1PCB1	L1PCB1	L1PCB1
13C12-PCB3	L1PCB3	I2PCB9	I2PCB9
PCB3	N1PCB3	L1PCB3	L1PCB3
PCB2	N1PCB2	L1PCB3	L1PCB_AVG
13C12-PCB4	L2PCB4	I2PCB9	I2PCB9
PCB4	N2PCB4	L2PCB4	L2PCB4
PCB10	N2PCB10	L2PCB4	L2PCB_AVG
PCB9	N2PCB9	L2PCB4	L2PCB_AVG
PCB7	N2PCB7	L2PCB4	L2PCB_AVG
PCB6	N2PCB6	L2PCB4	L2PCB_AVG
PCB5	N2PCB5	L2PCB4	L2PCB_AVG
13C12-PCB15	L2PCB15	I2PCB9	I2PCB9
PCB14	N2PCB14	L2PCB15	L2PCB_AVG
PCB11	N2PCB11	L2PCB15	L2PCB_AVG
PCB12+13	N2PCB12+13	L2PCB15	L2PCB_AVG
PCB15	N2PCB15	L2PCB15	L2PCB15
13C12-PCB19	L3PCB19	I2PCB9	I2PCB9
PCB19	N3PCB19	L3PCB19	L3PCB19
PCB18+30	N3PCB18+30	L3PCB19	L3PCB_AVG

PCB17	N3PCB17	L3PCB19	L3PCB_AVG
PCB27	N3PCB27	L3PCB19	L3PCB_AVG
PCB24	N3PCB24	L3PCB19	L3PCB_AVG
PCB16	N3PCB16	L3PCB19	L3PCB_AVG
PCB34	N3PCB34	L3PCB19	L3PCB_AVG
PCB23	N3PCB23	L3PCB19	L3PCB_AVG
PCB26+29	N3PCB26+29	L3PCB19	L3PCB_AVG
13C12-PCB8	LA2PCB8	I2PCB9	I2PCB9
PCB8	N2PCB8	LA2PCB8	LA2PCB8
13C12-PCB32	LA3PCB32	I2PCB9	I2PCB9
PCB32	N3PCB32	LA3PCB32	LA3PCB32
13C12-PCB31	LA3PCB31	I2PCB9	I2PCB9
PCB31	N3PCB31	LA3PCB31	LA3PCB31
13C12-PCB52	I4PCB52		
13C12-PCB37	L3PCB37	I4PCB52	I4PCB52
PCB25	N3PCB25	L3PCB37	L3PCB_AVG
PCB20+28	N3PCB20+28	L3PCB37	L3PCB_AVG
PCB21+33	N3PCB21+33	L3PCB37	L3PCB_AVG
PCB22	N3PCB22	L3PCB37	L3PCB_AVG
PCB36	N3PCB36	L3PCB37	L3PCB_AVG
PCB39	N3PCB39	L3PCB37	L3PCB_AVG
PCB38	N3PCB38	L3PCB37	L3PCB_AVG

Table 7 continued – PCB Congener Retention Time and Quantitation References

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
PCB35	N3PCB35	L3PCB37	L3PCB_AVG
PCB37	N3PCB37	L3PCB37	L3PCB37
13C12-PCB54	L4PCB54	I4PCB52	I4PCB52
PCB54	N4PCB54	L4PCB54	L4PCB54
PCB50+53	N4PCB50+53	L4PCB54	L4PCB_AVG
PCB45	N4PCB45	L4PCB54	L4PCB_AVG
PCB51	N4PCB51	L4PCB54	L4PCB_AVG
PCB46	N4PCB46	L4PCB54	L4PCB_AVG
PCB52	N4PCB52	L4PCB54	L4PCB_AVG
PCB73	N4PCB73	L4PCB54	L4PCB_AVG
PCB43	N4PCB43	L4PCB54	L4PCB_AVG
PCB49+69	N4PCB49+69	L4PCB54	L4PCB_AVG

PCB48	N4PCB48	L4PCB54	L4PCB_AVG
PCB59+62+75	N4PCB59+62+75	L4PCB54	L4PCB_AVG
PCB42	N4PCB42	L4PCB54	L4PCB_AVG
PCB41	N4PCB41	L4PCB54	L4PCB_AVG
PCB40+71	N4PCB40+71	L4PCB54	L4PCB_AVG
PCB64	N4PCB64	L4PCB54	L4PCB_AVG
13C12-PCB81	L4PCB81	I4PCB52	I4PCB52
PCB72	N4PCB72	L4PCB81	L4PCB_AVG
PCB68	N4PCB68	L4PCB81	L4PCB_AVG
PCB57	N4PCB57	L4PCB81	L4PCB_AVG
PCB58	N4PCB58	L4PCB81	L4PCB_AVG
PCB67	N4PCB67	L4PCB81	L4PCB_AVG
PCB63	N4PCB63	L4PCB81	L4PCB_AVG
PCB66	N4PCB66	L4PCB81	L4PCB_AVG
PCB55	N4PCB55	L4PCB81	L4PCB_AVG
PCB56	N4PCB56	L4PCB81	L4PCB_AVG
PCB80	N4PCB80	L4PCB81	L4PCB_AVG
PCB79	N4PCB79	L4PCB81	L4PCB_AVG
PCB78	N4PCB78	L4PCB81	L4PCB_AVG
PCB81	N4DLPCB81	L4PCB81	L4PCB81
13C12-PCB77	L4PCB77	I4PCB52	I4PCB52
PCB77	N4DLPCB77	L4PCB77	L4PCB77
13C12-PCB47	LA4PCB47	I4PCB52	I4PCB52
PCB44+47+65	N4PCB44+47+65	LA4PCB47	L4PCB_AVG
13C12-PCB70	LA4PCB70	I4PCB52	I4PCB52
PCB61+70+74+76	N4PCB61+70+74+76	LA4PCB70	L4PCB_AVG
13C12-PCB60	LA4PCB60	I4PCB52	I4PCB52
PCB60	N4PCB60	LA4PCB60	LA4PCB60
13C12-PCB28	C3PCB28	I4PCB52	I4PCB52
13C12-PCB101	I5PCB101		
13C12-PCB104	L5PCB104	I5PCB101	I5PCB101

Table 7 continued – PCB Congener Retention Time and Quantitation References

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
PCB104	N5PCB104	L5PCB104	L5PCB104
PCB96	N5PCB96	L5PCB104	L5PCB_AVG
PCB103	N5PCB103	L5PCB104	L5PCB_AVG

PCB94	N5PCB94	L5PCB104	L5PCB_AVG
PCB93+100	N5PCB93+100	L5PCB104	L5PCB_AVG
PCB98+102	N5PCB98+102	L5PCB104	L5PCB_AVG
PCB88	N5PCB88	L5PCB104	L5PCB_AVG
PCB91	N5PCB91	L5PCB104	L5PCB_AVG
PCB84	N5PCB84	L5PCB104	L5PCB_AVG
PCB89	N5PCB89	L5PCB104	L5PCB_AVG
PCB121	N5PCB121	L5PCB104	L5PCB_AVG
13C12-PCB123	L5PCB123	I5PCB101	I5PCB101
PCB92	N5PCB92	L5PCB123	L5PCB_AVG
PCB90+101+113	N5PCB90+101+113	L5PCB123	L5PCB_AVG
PCB83	N5PCB83	L5PCB123	L5PCB_AVG
PCB99	N5PCB99	L5PCB123	L5PCB_AVG
PCB112	N5PCB112	L5PCB123	L5PCB_AVG
PCB86+87+97+109+119+125	N5PCB86+87+97+109+119+125	L5PCB123	L5PCB_AVG
PCB110+115	N5PCB110+115	L5PCB123	L5PCB_AVG
PCB82	N5PCB82	L5PCB123	L5PCB_AVG
PCB111	N5PCB111	L5PCB123	L5PCB_AVG
PCB120	N5PCB120	L5PCB123	L5PCB_AVG
PCB108+124	N5PCB108+124	L5PCB123	L5PCB_AVG
PCB107	N5PCB107	L5PCB123	L5PCB_AVG
PCB123	N5DLPCB123	L5PCB123	L5PCB123
PCB106	N5PCB106	L5PCB123	L5PCB_AVG
13C12-PCB118	L5PCB118	I5PCB101	I5PCB101
PCB118	N5DLPCB118	L5PCB118	L5PCB118
PCB122	N5PCB122	L5PCB118	L5PCB_AVG
13C12-PCB114	L5PCB114	I5PCB101	I5PCB101
PCB114	N5DLPCB114	L5PCB114	L5PCB114
13C12-PCB105	L5PCB105	I5PCB101	I5PCB101
PCB105	N5DLPCB105	L5PCB105	L5PCB105
13C12-PCB126	L5PCB126	I5PCB101	I5PCB101
PCB126	N5DLPCB126	L5PCB126	L5PCB126
13C12-PCB95	LA5PCB95	I5PCB101	I5PCB101
PCB95	N5PCB95	LA5PCB95	LA5PCB95
13C12-PCB85	LA5PCB85	I5PCB101	I5PCB101
PCB85+116+117	N5PCB85+116+117	LA5PCB85	L5PCB_AVG
13C12-PCB127	LA5PCB127	I5PCB101	I5PCB101
PCB127	N5PCB127	LA5PCB127	LA5PCB127
13C12-PCB111	C5PCB111	I5PCB101	I5PCB101
13C12-PCB138	I6PCB138		

Table 7 continued – PCB Congener Retention Time and Quantitation References

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
13C12-PCB155	L6PCB155	I6PCB138	I6PCB138
PCB155	N6PCB155	L6PCB155	L6PCB155
PCB152	N6PCB152	L6PCB155	L6PCB_AVG
PCB150	N6PCB150	L6PCB155	L6PCB_AVG
PCB136	N6PCB136	L6PCB155	L6PCB_AVG
PCB145	N6PCB145	L6PCB155	L6PCB_AVG
PCB148	N6PCB148	L6PCB155	L6PCB_AVG
PCB154	N6PCB154	L6PCB155	L6PCB_AVG
PCB135+151	N6PCB135+151	L6PCB155	L6PCB_AVG
PCB144	N6PCB144	L6PCB155	L6PCB_AVG
PCB147+149	N6PCB147+149	L6PCB155	L6PCB_AVG
PCB134	N6PCB134	L6PCB155	L6PCB_AVG
PCB143	N6PCB143	L6PCB155	L6PCB_AVG
PCB139+140	N6PCB139+140	L6PCB155	L6PCB_AVG
PCB131	N6PCB131	L6PCB155	L6PCB_AVG
PCB142	N6PCB142	L6PCB155	L6PCB_AVG
PCB132	N6PCB132	L6PCB155	L6PCB_AVG
13C12-PCB167	L6PCB167	I6PCB138	I6PCB138
PCB165	N6PCB165	L6PCB167	L6PCB_AVG
PCB146	N6PCB146	L6PCB167	L6PCB_AVG
PCB161	N6PCB161	L6PCB167	L6PCB_AVG
PCB153+168	N6PCB153+168	L6PCB167	L6PCB_AVG
PCB130	N6PCB130	L6PCB167	L6PCB_AVG
PCB137	N6PCB137	L6PCB167	L6PCB_AVG
PCB164	N6PCB164	L6PCB167	L6PCB_AVG
PCB160	N6PCB160	L6PCB167	L6PCB_AVG
PCB129+138+163	N6PCB129+138+163	L6PCB167	L6PCB_AVG
PCB158	N6PCB158	L6PCB167	L6PCB_AVG
PCB159	N6PCB159	L6PCB167	L6PCB_AVG
PCB167	N6DLPCB167	L6PCB167	L6PCB167
13C12-PCB156+157	L6PCB156+157	I6PCB138	I6PCB138
PCB156+157	N6DLPCB156+157	L6PCB156+157	L6PCB156+157
13C12-PCB169	L6PCB169	I6PCB138	I6PCB138
PCB169	N6DLPCB169	L6PCB169	L6PCB169

13C12-PCB133	LA6PCB133	I6PCB138	I6PCB138
PCB133	N6PCB133	LA6PCB133	LA6PCB133
13C12-PCB141	LA6PCB141	I6PCB138	I6PCB138
PCB141	N6PCB141	LA6PCB141	LA6PCB141
13C12-PCB128	LA6PCB128	I6PCB138	I6PCB138
PCB128+166	N6PCB128+166	LA6PCB128	L6PCB_AVG
13C12-PCB162	LA6PCB162	I6PCB138	I6PCB138
PCB162	N6PCB162	LA6PCB162	LA6PCB162
13C12-PCB178	C7PCB178	I6PCB138	I6PCB138

Table 7 continued – PCB Congener Retention Time and Quantitation References

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
13C12-PCB194	I8PCB194		
13C12-PCB188	L7PCB188	I8PCB194	I8PCB194
PCB188	N7PCB188	L7PCB188	L7PCB188
PCB179	N7PCB179	L7PCB188	L7PCB_AVG
PCB184	N7PCB184	L7PCB188	L7PCB_AVG
PCB176	N7PCB176	L7PCB188	L7PCB_AVG
PCB186	N7PCB186	L7PCB188	L7PCB_AVG
PCB178	N7PCB178	L7PCB188	L7PCB_AVG
PCB175	N7PCB175	L7PCB188	L7PCB_AVG
PCB187	N7PCB187	L7PCB188	L7PCB_AVG
PCB182	N7PCB182	L7PCB188	L7PCB_AVG
PCB183+185	N7PCB183+185	L7PCB188	L7PCB_AVG
PCB174	N7PCB174	L7PCB188	L7PCB_AVG
PCB177	N7PCB177	L7PCB188	L7PCB_AVG
PCB181	N7PCB181	L7PCB188	L7PCB_AVG
PCB171+173	N7PCB171+173	L7PCB188	L7PCB_AVG
13C12-PCB189	L7PCB189	I8PCB194	I8PCB194
PCB172	N7PCB172	L7PCB189	L7PCB_AVG
PCB192	N7PCB192	L7PCB189	L7PCB_AVG
PCB191	N7PCB191	L7PCB189	L7PCB_AVG
PCB170	N7PCB170	L7PCB189	L7PCB_AVG
PCB190	N7PCB190	L7PCB189	L7PCB_AVG
PCB189	N7DLPCB189	L7PCB189	L7PCB189
13C12-PCB202	L8PCB202	I8PCB194	I8PCB194
PCB202	N8PCB202	L8PCB202	L8PCB202
PCB201	N8PCB201	L8PCB202	L8PCB_AVG
PCB204	N8PCB204	L8PCB202	L8PCB_AVG

PCB197+200	N8PCB197+200	L8PCB202	L8PCB_AVG
PCB198+199	N8PCB198+199	L8PCB202	L8PCB_AVG
13C12-PCB205	L8PCB205	I8PCB194	I8PCB194
PCB196	N8PCB196	L8PCB205	L8PCB_AVG
PCB203	N8PCB203	L8PCB205	L8PCB_AVG
PCB195	N8PCB195	L8PCB205	L8PCB_AVG
PCB194	N8PCB194	L8PCB205	L8PCB_AVG
PCB205	N8PCB205	L8PCB205	L8PCB205
13C12-PCB208	L9PCB208	I8PCB194	I8PCB194
PCB208	N9PCB208	L9PCB208	L9PCB208
PCB207	N9PCB207	L9PCB208	L9PCB_AVG
13C12-PCB206	L9PCB206	I8PCB194	I8PCB194
PCB206	N9PCB206	L9PCB206	L9PCB206
13C12-PCB209	L10PCB209	I8PCB194	I8PCB194
PCB209	N10PCB209	L10PCB209	L10PCB209
13C12-PCB180	LA7PCB180	I8PCB194	I8PCB194
PCB180+193	N7PCB180+193	LA7PCB180	L7PCB_AVG

Table 7A

PCB Congener RT and Quantitation References Tox/LOC ICAL

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
13C12-PCB9	I2PCB9		
13C12-PCB1	L1PCB1	I2PCB9	I2PCB9
PCB1	N1PCB1	L1PCB1	L1PCB1
13C12-PCB3	L1PCB3	I2PCB9	I2PCB9
PCB2	N1PCB2	L1PCB3	L1PCB_AVG
PCB3	N1PCB3	L1PCB3	L1PCB3
13C12-PCB4	L2PCB4	I2PCB9	I2PCB9
PCB4	N2PCB4	L2PCB4	L2PCB4
PCB10	N2PCB10	L2PCB4	L2PCB_AVG
PCB9	N2PCB9	L2PCB4	L2PCB_AVG
PCB7	N2PCB7	L2PCB4	L2PCB_AVG
PCB6	N2PCB6	L2PCB4	L2PCB_AVG
PCB5	N2PCB5	L2PCB4	L2PCB_AVG
PCB8	N2PCB8	L2PCB4	L2PCB_AVG
13C12-PCB15	L2PCB15	I2PCB9	I2PCB9

PCB14	N2PCB14	L2PCB15	L2PCB_AVG
PCB11	N2PCB11	L2PCB15	L2PCB_AVG
PCB12+13	N2PCB12+13	L2PCB15	L2PCB_AVG
PCB15	N2PCB15	L2PCB15	L2PCB15
13C12-PCB19	L3PCB19	I2PCB9	I2PCB9
PCB19	N3PCB19	L3PCB19	L3PCB19
PCB18+30	N3PCB18+30	L3PCB19	L3PCB_AVG
PCB17	N3PCB17	L3PCB19	L3PCB_AVG
PCB27	N3PCB27	L3PCB19	L3PCB_AVG
PCB24	N3PCB24	L3PCB19	L3PCB_AVG
PCB16	N3PCB16	L3PCB19	L3PCB_AVG
PCB32	N3PCB32	L3PCB19	L3PCB_AVG
PCB34	N3PCB34	L3PCB19	L3PCB_AVG
PCB23	N3PCB23	L3PCB19	L3PCB_AVG
PCB26+29	N3PCB26+29	L3PCB19	L3PCB_AVG
13C12-PCB52	I4PCB52		
13C12-PCB37	L3PCB37	I4PCB52	I4PCB52
PCB25	N3PCB25	L3PCB37	L3PCB_AVG
PCB31	N3PCB31	L3PCB37	L3PCB_AVG
PCB20+28	N3PCB20+28	L3PCB37	L3PCB_AVG
PCB21+33	N3PCB21+33	L3PCB37	L3PCB_AVG
PCB22	N3PCB22	L3PCB37	L3PCB_AVG
PCB36	N3PCB36	L3PCB37	L3PCB_AVG
PCB39	N3PCB39	L3PCB37	L3PCB_AVG
PCB38	N3PCB38	L3PCB37	L3PCB_AVG
PCB35	N3PCB35	L3PCB37	L3PCB_AVG
PCB37	N3PCB37	L3PCB37	L3PCB37
13C12-PCB54	L4PCB54	I4PCB52	I4PCB52

Table 7A continued – PCB Congener RT and Quantitation References Tox/LOC ICAL

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
PCB54	N4PCB54	L4PCB54	L4PCB54
PCB50+53	N4PCB50+53	L4PCB54	L4PCB_AVG
PCB45	N4PCB45	L4PCB54	L4PCB_AVG
PCB51	N4PCB51	L4PCB54	L4PCB_AVG
PCB46	N4PCB46	L4PCB54	L4PCB_AVG
PCB52	N4PCB52	L4PCB54	L4PCB_AVG

PCB73	N4PCB73	L4PCB54	L4PCB_AVG
PCB43	N4PCB43	L4PCB54	L4PCB_AVG
PCB49+69	N4PCB49+69	L4PCB54	L4PCB_AVG
PCB48	N4PCB48	L4PCB54	L4PCB_AVG
PCB44+47+65	N4PCB44+47+65	L4PCB54	L4PCB_AVG
PCB59+62+75	N4PCB59+62+75	L4PCB54	L4PCB_AVG
PCB42	N4PCB42	L4PCB54	L4PCB_AVG
PCB41	N4PCB41	L4PCB54	L4PCB_AVG
PCB40+71	N4PCB40+71	L4PCB54	L4PCB_AVG
PCB64	N4PCB64	L4PCB54	L4PCB_AVG
13C12-PCB81	L4PCB81	I4PCB52	I4PCB52
PCB72	N4PCB72	L4PCB81	L4PCB_AVG
PCB68	N4PCB68	L4PCB81	L4PCB_AVG
PCB57	N4PCB57	L4PCB81	L4PCB_AVG
PCB58	N4PCB58	L4PCB81	L4PCB_AVG
PCB67	N4PCB67	L4PCB81	L4PCB_AVG
PCB63	N4PCB63	L4PCB81	L4PCB_AVG
PCB61+70+74+76	N4PCB61+70+74+76	L4PCB81	L4PCB_AVG
PCB66	N4PCB66	L4PCB81	L4PCB_AVG
PCB55	N4PCB55	L4PCB81	L4PCB_AVG
PCB56	N4PCB56	L4PCB81	L4PCB_AVG
PCB60	N4PCB60	L4PCB81	L4PCB_AVG
PCB80	N4PCB80	L4PCB81	L4PCB_AVG
PCB79	N4PCB79	L4PCB81	L4PCB_AVG
PCB78	N4PCB78	L4PCB81	L4PCB_AVG
PCB81	N4DLPCB81	L4PCB81	L4PCB81
13C12-PCB77	L4PCB77	I4PCB52	I4PCB52
PCB77	N4DLPCB77	L4PCB77	L4PCB77
13C12-PCB28	C3PCB28	I4PCB52	I4PCB52
13C12-PCB101	I5PCB101		
13C12-PCB104	L5PCB104	I5PCB101	I5PCB101
PCB104	N5PCB104	L5PCB104	L5PCB104
PCB96	N5PCB96	L5PCB104	L5PCB_AVG
PCB103	N5PCB103	L5PCB104	L5PCB_AVG
PCB94	N5PCB94	L5PCB104	L5PCB_AVG
PCB95	N5PCB95	L5PCB104	L5PCB_AVG
PCB93+100	N5PCB93+100	L5PCB104	L5PCB_AVG

Table 7A continued – PCB Congener RT and Quantitation References Tox/LOC ICAL

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
PCB98+102	N5PCB98+102	L5PCB104	L5PCB_AVG
PCB88	N5PCB88	L5PCB104	L5PCB_AVG
PCB91	N5PCB91	L5PCB104	L5PCB_AVG
PCB84	N5PCB84	L5PCB104	L5PCB_AVG
PCB89	N5PCB89	L5PCB104	L5PCB_AVG
PCB121	N5PCB121	L5PCB104	L5PCB_AVG
13C12-PCB123	L5PCB123	I5PCB101	I5PCB101
PCB92	N5PCB92	L5PCB123	L5PCB_AVG
PCB90+101+113	N5PCB90+101+113	L5PCB123	L5PCB_AVG
PCB83	N5PCB83	L5PCB123	L5PCB_AVG
PCB99	N5PCB99	L5PCB123	L5PCB_AVG
PCB112	N5PCB112	L5PCB123	L5PCB_AVG
PCB86+87+97+109+119+125	N5PCB86+87+97+109+119+125	L5PCB123	L5PCB_AVG
PCB85+116+117	N5PCB85+116+117	L5PCB123	L5PCB_AVG
PCB110+115	N5PCB110+115	L5PCB123	L5PCB_AVG
PCB82	N5PCB82	L5PCB123	L5PCB_AVG
PCB111	N5PCB111	L5PCB123	L5PCB_AVG
PCB120	N5PCB120	L5PCB123	L5PCB_AVG
PCB108+124	N5PCB108+124	L5PCB123	L5PCB_AVG
PCB107	N5PCB107	L5PCB123	L5PCB_AVG
PCB123	N5DLPCB123	L5PCB123	L5PCB123
PCB106	N5PCB106	L5PCB123	L5PCB_AVG
13C12-PCB118	L5PCB118	I5PCB101	I5PCB101
PCB118	N5DLPCB118	L5PCB118	L5PCB118
PCB122	N5PCB122	L5PCB118	L5PCB_AVG
13C12-PCB114	L5PCB114	I5PCB101	I5PCB101
PCB114	N5DLPCB114	L5PCB114	L5PCB114
13C12-PCB105	L5PCB105	I5PCB101	I5PCB101
PCB105	N5DLPCB105	L5PCB105	L5PCB105
PCB127	N5PCB127	L5PCB105	L5PCB_AVG
13C12-PCB126	L5PCB126	I5PCB101	I5PCB101
PCB126	N5DLPCB126	L5PCB126	L5PCB126
13C12-PCB111	C5PCB111	I5PCB101	I5PCB101
13C12-PCB138	I6PCB138		
13C12-PCB155	L6PCB155	I6PCB138	I6PCB138
PCB155	N6PCB155	L6PCB155	L6PCB155
PCB152	N6PCB152	L6PCB155	L6PCB_AVG
PCB150	N6PCB150	L6PCB155	L6PCB_AVG

PCB136	N6PCB136	L6PCB155	L6PCB_AVG
PCB145	N6PCB145	L6PCB155	L6PCB_AVG
PCB148	N6PCB148	L6PCB155	L6PCB_AVG
PCB154	N6PCB154	L6PCB155	L6PCB_AVG
PCB135+151	N6PCB135+151	L6PCB155	L6PCB_AVG

Table 7A continued – PCB Congener RT and Quantitation References Tox/LOC ICAL

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
PCB144	N6PCB144	L6PCB155	L6PCB_AVG
PCB147+149	N6PCB147+149	L6PCB155	L6PCB_AVG
PCB134	N6PCB134	L6PCB155	L6PCB_AVG
PCB143	N6PCB143	L6PCB155	L6PCB_AVG
PCB139+140	N6PCB139+140	L6PCB155	L6PCB_AVG
PCB131	N6PCB131	L6PCB155	L6PCB_AVG
PCB142	N6PCB142	L6PCB155	L6PCB_AVG
PCB132	N6PCB132	L6PCB155	L6PCB_AVG
PCB133	N6PCB133	L6PCB155	L6PCB_AVG
13C12-PCB167	L6PCB167	I6PCB138	I6PCB138
PCB165	N6PCB165	L6PCB167	L6PCB_AVG
PCB146	N6PCB146	L6PCB167	L6PCB_AVG
PCB161	N6PCB161	L6PCB167	L6PCB_AVG
PCB153+168	N6PCB153+168	L6PCB167	L6PCB_AVG
PCB141	N6PCB141	L6PCB167	L6PCB_AVG
PCB130	N6PCB130	L6PCB167	L6PCB_AVG
PCB137	N6PCB137	L6PCB167	L6PCB_AVG
PCB164	N6PCB164	L6PCB167	L6PCB_AVG
PCB160	N6PCB160	L6PCB167	L6PCB_AVG
PCB129+138+163	N6PCB129+138+163	L6PCB167	L6PCB_AVG
PCB158	N6PCB158	L6PCB167	L6PCB_AVG
PCB128+166	N6PCB128+166	L6PCB167	L6PCB_AVG
PCB159	N6PCB159	L6PCB167	L6PCB_AVG
PCB162	N6PCB162	L6PCB167	L6PCB_AVG
PCB167	N6DLPCB167	L6PCB167	L6PCB167
13C12-PCB156+157	L6PCB156+157	I6PCB138	I6PCB138
PCB156+157	N6DLPCB156+157	L6PCB156+157	L6PCB156+157
13C12-PCB169	L6PCB169	I6PCB138	I6PCB138
PCB169	N6DLPCB169	L6PCB169	L6PCB169

13C12-PCB178	C7PCB178	I6PCB138	I6PCB138
13C12-PCB194	I8PCB194		
13C12-PCB188	L7PCB188	I8PCB194	I8PCB194
PCB188	N7PCB188	L7PCB188	L7PCB188
PCB179	N7PCB179	L7PCB188	L7PCB_AVG
PCB184	N7PCB184	L7PCB188	L7PCB_AVG
PCB176	N7PCB176	L7PCB188	L7PCB_AVG
PCB186	N7PCB186	L7PCB188	L7PCB_AVG
PCB178	N7PCB178	L7PCB188	L7PCB_AVG
PCB175	N7PCB175	L7PCB188	L7PCB_AVG
PCB187	N7PCB187	L7PCB188	L7PCB_AVG
PCB182	N7PCB182	L7PCB188	L7PCB_AVG
PCB183+185	N7PCB183+185	L7PCB188	L7PCB_AVG
PCB174	N7PCB174	L7PCB188	L7PCB_AVG

Table 7A continued – PCB Congener RT and Quantitation References Tox/LOC ICAL

Compound Name	Entry Identifier	Retention Time Identifier	Quantitation Reference
PCB177	N7PCB177	L7PCB188	L7PCB_AVG
PCB181	N7PCB181	L7PCB188	L7PCB_AVG
PCB171+173	N7PCB171+173	L7PCB188	L7PCB_AVG
13C12-PCB189	L7PCB189	I8PCB194	I8PCB194
PCB172	N7PCB172	L7PCB189	L7PCB_AVG
PCB192	N7PCB192	L7PCB189	L7PCB_AVG
PCB180+193	N7PCB180+193	L7PCB189	L7PCB_AVG
PCB191	N7PCB191	L7PCB189	L7PCB_AVG
PCB170	N7PCB170	L7PCB189	L7PCB_AVG
PCB190	N7PCB190	L7PCB189	L7PCB_AVG
PCB189	N7DLPCB189	L7PCB189	L7PCB189
13C12-PCB202	L8PCB202	I8PCB194	I8PCB194
PCB202	N8PCB202	L8PCB202	L8PCB202
PCB201	N8PCB201	L8PCB202	L8PCB_AVG
PCB204	N8PCB204	L8PCB202	L8PCB_AVG
PCB197+200	N8PCB197+200	L8PCB202	L8PCB_AVG
PCB198+199	N8PCB198+199	L8PCB202	L8PCB_AVG
13C12-PCB205	L8PCB205	I8PCB194	I8PCB194
PCB196	N8PCB196	L8PCB205	L8PCB_AVG
PCB203	N8PCB203	L8PCB205	L8PCB_AVG

PCB195	N8PCB195	L8PCB205	L8PCB_AVG
PCB194	N8PCB194	L8PCB205	L8PCB_AVG
PCB205	N8PCB205	L8PCB205	L8PCB205
13C12-PCB208	L9PCB208	I8PCB194	I8PCB194
PCB208	N9PCB208	L9PCB208	L9PCB208
PCB207	N9PCB207	L9PCB208	L9PCB_AVG
13C12-PCB206	L9PCB206	I8PCB194	I8PCB194
PCB206	N9PCB206	L9PCB206	L9PCB206
13C12-PCB209	L10PCB209	I8PCB194	I8PCB194
PCB209	N10PCB209	L10PCB209	L10PCB209

Table 8

Labeled PCB Congener Average Quantitation Reference

Average Quantitation Reference Name	Entry Identifier	Quantitation Reference
AVG_L1PCB	L1PCB_AVG	I2PCB9 AVG(L1PCB1 / L1PCB3)
AVG_L2PCB	L2PCB_AVG	I2PCB9 AVG(L2PCB4 / L2PCB15 / LA2PCB8)
AVG_L3PCB	L3PCB_AVG	I2PCB9 AVG(L3PCB19 / L3PCB37 / LA3PCB32 / LA3PCB31)
AVG_L4PCB	L4PCB_AVG	I4PCB52 AVG(L4PCB54 / L4PCB81 / L4PCB77 / LA4PCB47 / LA4PCB70 / LA4PCB60)
AVG_L5PCB	L5PCB_AVG	I5PCB101 AVG(L5PCB104 / L5PCB123 / L5PCB114 / L5PCB118 / L5PCB105 / LA5PCB95
AVG_L6PCB	L6PCB_AVG	I6PCB138 AVG(L6PCB155 / L6PCB156+157 / L6PCB167 / L6PCB169 / LA6PCB133 / LA6F
AVG_L7PCB	L7PCB_AVG	I8PCB194 AVG(L7PCB188 / L7PCB189 / LA7PCB180)
AVG_L8PCB	L8PCB_AVG	I8PCB194 AVG(L8PCB202 / L8PCB205)
AVG_L9PCB	L9PCB_AVG	I8PCB194 AVG(L9PCB208 / L9PCB206)

Table 8A

Labeled PCB Congener Average Quantitation Reference Tox/LOC ICAL

Average Quantitation Reference Name	Entry Identifier	Quantitation Reference
AVG_L1PCB	L1PCB_AVG	I2PCB9 AVG(L1PCB1 / L1PCB3)
AVG_L2PCB	L2PCB_AVG	I2PCB9 AVG(L2PCB4 / L2PCB15)
AVG_L3PCB	L3PCB_AVG	I2PCB9 AVG(L3PCB19 / L3PCB37)

AVG_L4PCB	L4PCB_AVG	I4PCB52 AVG(L4PCB54 / L4PCB81 / L4PCB77)
AVG_L5PCB	L5PCB_AVG	I5PCB101 AVG(L5PCB104 / L5PCB123 / L5PCB114 / L5PCB118 / L5PCB105)
AVG_L6PCB	L6PCB_AVG	I6PCB138 AVG(L6PCB155 / L6PCB156+157 / L6PCB167 / L6PCB169)
AVG_L7PCB	L7PCB_AVG	I8PCB194 AVG(L7PCB188 / L7PCB189)
AVG_L8PCB	L8PCB_AVG	I8PCB194 AVG(L8PCB202 / L8PCB205)
AVG_L9PCB	L9PCB_AVG	I8PCB194 AVG(L9PCB208 / L9PCB206)

QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
T-HRMS-WI12032 Separatory Funnel Extraction Procedure for HRMS Analysis in an Aqueous Matrix Using Method 1613B, 8290A, 1668A, and 1668C
T-HRMS-WI19229 DFS HRGC/HRMS Preventative and Corrective Maintenance
T-HRMS-WI9433 Processing High Resolution Mass Spectrometry Data Using TargetQuan
T-HRMS-WI9446 Standards Management in the High Resolution Mass Spectrometry Laboratory
T-HRMS-WI9488 Soxhlet Extraction Procedure for HRMS Analysis in a Solid Matrix by Methods: 1613B, 8290A, 1668C, and 1668A

End of document

Version history

Version	Approval	Revision information
6	27.JAN.2016	
7	16.MAY.2018	

BR-GC-005

Polychlorinated Biphenyls (PCBs) by GC/ECD (SW846 8082A, USEPA TO4A/TO10A (Rev. 13.0, 01/24/18)

Title: Polychlorinated Biphenyls (PCBs) by GC/ECD (SW846 8082A, USEPA TO4A/TO10A)

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1.0 Scope and Application

This SOP describes the laboratory procedure used to determine the concentration of polychlorinated biphenyls (PCBs) as Aroclors using dual column gas chromatography with electron capture detectors (GC/ECD).

This SOP is applicable to instrument analysis only. Extraction and extract cleanup procedures are provided in separate SOPs.

1.1 Analytes, Matrices, and Reporting Limits

This procedure may be used for a variety of matrices including: air, sediment, soil, tissue, and water.

The list of target compounds that can be determined from this method along with the associated reporting limits (RL) is provided in Table 1.

2.0 Summary of Method

2.0uL of extract is injected into a dual capillary column gas chromatograph equipped with electron capture detectors (GC/ECD). The chromatographic data is used to determine the list of analytes provided in Table 1. NOTE: Injection volumes used must be validated and all samples are to be analyzed with the same injection volume used for standard solutions within the initial calibration. Quantitation is accomplished by averaging the concentration of three to five peaks for each Aroclor against a calibration curve. Internal standards are used to compensate for instrument variation.

This SOP is based on the following reference methods:

- SW-846 Method 8082A Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, February 2007.
- Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999 (EPA/625/R-96/010b)
- Compendium Method TO-10A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999 (EPA/625/R-96/010b)

If the laboratory procedure is modified from the above reference method, a list of modifications will be provided in Section 16.0 of this SOP.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

- Method interference may be caused by contaminants in the extraction solvent. Solvents should be stored away from organochlorine compounds to minimize contamination.

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- Non-target compounds co-extracted from the sample matrix can also cause interference, the extent of which will vary depending on the nature of the samples. Elemental sulfur is often found in sediment samples and its presence will result in broad peaks. Samples are screened prior to analysis, and those samples that contain high levels of sulfur are subject to sulfur cleanup (SW-846 3660B). Cleanup procedures that may be used for this method include: GPC (SW-846-3640A), silica gel (SW-846 3630C), Florisil (SW-846 3620B), and Sulfuric acid Cleanup (SW-846 3665A).

- Phthalate esters introduced during sample preparation can pose a problem in the determination of target analytes. Common flexible plastics contain varying amounts of phthalate esters. These phthalate esters can be easily extracted or leached during extraction. To minimize this interference, avoid contact with any plastic materials.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst must be aware of the locations of those zones and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off or disconnect it from its source of power.

5.2 Primary Materials Used

Table 2 lists materials used in this method which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Miscellaneous

- Autosampler Vials, National Scientific or equivalent.

- Hydrogen Generator: Parker Hannafin H2-1200 or equivalent.

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- Volumetric Syringes, Class "A" (10µl, 25µl, 50µl, 100µl, 250µl and 500µl), Hamilton or equivalent.

6.2 Analytical System

- Computer Hardware/Software: GC Acquisition Platform – ChemStation. Data Processing – TestAmerica Chrom, current version.

- GC/ECD: with dual columns, dual ECDs, and auto-sampler capable of injecting a 0.2-2.0 µL aliquot split onto two columns: Agilent Technologies 6890N with 7683 Series injector, or equivalent.

- GC Columns: A dual fused silica capillary column system that will provide simultaneous primary and confirmation analyses:
  - RTX-CLP, (30m x 0.32 mm ID x 0.5µm)
  - RTX-CLPII, (30m x 0.25 mm ID x 0.5µm)

Equivalent columns may be used provided the elution orders are documented and compound separations are maintained.

7.0 Reagents and Standards

7.1 Reagents

- Hexane, Ultra-Resi Analyzed, JT Baker or equivalent.

7.2 Standards

Purchase stock standard solutions from commercial vendors and from these prepare calibration and working standards by diluting a known volume of stock standard in an appropriate solvent to the final volume needed to achieve the desired concentration. The recommended formulation for each standard used in this procedure is provided in Appendix B along with the recommended source materials, expiration dates and storage conditions.

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection, so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method. Listed below are minimum sample size, preservation, and holding time requirements needed for this test.

Matrix	Sample Container	Minimum Sample Size	Preservation	Extract Holding Time	Reference
Air	Polyurethane Foam Plug (PUF)	1 PUF	Chilled to 4℃	40 Days	TO-10A
Air	PUF	1 PUF	Chilled to 4℃	40 Days	TO-4A
Water	Glass	1 L	Chilled to 4℃	40 Days	SW-846 8082A

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Solid	Glass	50 g	Chilled to 4℃	40 Days	SW-846 8082A

Analytical holding time is determined from date of initiation of extraction.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

Note: EPA SW-846 Update IV Chapter 4 re-defined the holding times for PCBs as "NONE". The EPA Methods Update Rule re-defined holding times for PCBs as one year from collection to extraction and one year from extraction to analysis. The laboratory incorporates the extended preparation holding times for PCBs on a project-specific basis, as allowed by that particular state agency and client.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer field samples	See Table 3
Laboratory Control Sample (LCS)	1 in 20 or fewer field samples	See Table 3
Laboratory Control Sample Duplicate (LCSD)	1 in 20 or fewer field samples, if volume is not present for MS/MSD	See Table 3
Matrix Spike(s) MS/MSD	1 in 20 or fewer field samples	See Table 3
Sample Duplicate (SD)	Client Request	See Table 3

9.2 Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Table 3
Second Source Calibration Verification (ICV)	Once, after each ICAL	See Table 3
Continuing Calibration Verification (CCV)	Daily, every 20 field samples or 12 hours (whichever is sooner)	See Table 3
Retention Time Windows	Once every 24 hours for all Aroclors reported (8082A). Once every 24 hours for Aroclor 1016/1260 (TO4/TO10)	See Table 3

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10.0 Procedure

10.1 Instrument Operating Conditions

Install a five meter deactivated guard column into the injection port and connect the guard column to the separate analytical columns using a glass "Y". The analytical columns are installed into independent ECD detectors.

The recommended instrument operating conditions are as follows:

Initial Temperature	130ºC
Temperature Program	30ºC per minute to 210ºC; 10ºC per minute to 250ºC, hold 0.37 minutes; 20.0ºC per minute to 300°C. 30ºC per minute to 320 ºC. Hold for 3 minutes.
Detector Temperature	300ºC
Injector Temperature	220ºC
Injection volume	2.0µL
Carrier Gas	Hydrogen

Optimize the flow rate of the carrier gas by injecting an un-retained substance onto the column at an isothermal oven state and adjusting the flow to obtain the recommended dead volume time.

10.2 Retention Time Window Establishment

Whenever a new GC column is installed, establish RT windows for each analyte by analyzing three standards over a 72-hour period. Calculate the mean RT and Standard Deviation (SD). The RT window is calculated as the mean RT ± 3SD. If the SD is <0.01 minutes, a default SD of 0.01 minutes may be used.

If this procedure results in RT windows that are too tight, favoring false negatives, the laboratory may opt to use an alternate method to determine the RT windows. An alternate method consists of using a RT window of ± 0.05 minutes. The center of the RT window is set at the midpoint calibration level in the initial calibration sequence. RT windows are then updated daily (minimum frequency), re-centering the windows on the retention times established in a CCV.

When determining retention time windows, analyze a standard of DDT to ensure that the aroclor peaks being used do not interfere with the major single-peak pesticides (DDT, DDE, DDD).

10.3 Instrument Calibration

10.3.1 Initial Calibration (ICAL)

Analyze a minimum of five calibration levels for each Aroclor. The following Aroclors may be combined in the standards: 1016/1260 (also known as 1660), 1221/1254, 1232/1262, 1242/1268. Aroclor 1248 may not be combined with other Aroclors.

Tetrachloro-m-xylene and Decachlorbiphenyl are calibrated from the Aroclor 1660 standards.

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Note: If the calibration will be used only for 8082A analysis, a single point calibration may be used except for Aroclors 1016 and 1260. The full set of Aroclors do not need to be calibrated if used for projects with a limited requested list. Every initial calibration block requires at least one standard with Aroclor 1016 to be analyzed for pattern recognition purposes, however.

Prepare the calibration standards using the formulations provided in Appendix B then transfer 100 µg/L to an autosampler vial insert. Add 4uL of the internal standard (IS) solution, then crimp the vial tightly.

Place the vials in the autosampler, and initiate the analytical sequence.

A minimum of five quantitation peaks must be chosen for each Aroclor with the exception of Aroclor 1221 which requires a minimum of 3 peaks. The peaks must be characteristic of the Aroclor in question.

The peaks used for quantitation in each Aroclor standard must be at least 25% of the height of the largest Aroclor peak with the exception of Aroclor 1268 where the requirement is 10%.

Each set of 5 peaks should include at least one peak that is unique to that Aroclor.

Calculate the Calibration Factor (CF), mean CF, and Percent Relative Standard Deviation (%RSD) for each analyte on both columns using the peak height, adjusted for internal standard response.

The %RSD for each target analyte must be less than or equal to 20% in order to use the mean CF for quantification. This evaluation is performed for each quantitation peak chosen for each Aroclor. All peaks must individually pass the 20% evaluation.

Alternately, use linear regression with a correlation coefficient (r) ≥ 0.995. Linear regression requires a minimum of five calibration points.

The mid-level AR1660 ICAL standard must meet a resolution criteria of <75% for the triplet near the end of the AR1260 pattern on at least one column. The resolution may be calculated per the equation in Appendix C.

10.3.2 Second Source Calibration Verification (ICV)

Immediately after each calibration and prior to the analysis of any QC or field samples, verify the accuracy of the initial calibration by analyzing a standard prepared from a second source of Aroclor 1660.

Prepare the ICV using the formulation provided in Appendix B. Aliquot 100uL of the working standard into a vial, and add 4uL of the IS solution. Crimp tightly. Inject the ICV standard onto the instrument in the same manner as performed for the initial calibration standards.

The percent recovery of the must be within ± 20% of the expected value of the average concentration of each Aroclor.

If this criterion is not met, correct the problem and reanalyze the ICV. If reanalysis fails, remake the calibration standards and/or perform instrument maintenance and recalibrate. The acceptance criteria must be met on both columns.

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10.3.3 Continuing Calibration Verification (CCV)

Analyze a CCV (AR1660) at or below the mid-calibration range each day before sample analysis. This CCV is used to update the retention times of AR1660.

Analyze a CCV (AR1660) at least every 20 field samples for 8082A. If Aroclors other than 1660 are identified in a sample, a CCV of the identified Aroclor must be analyzed within 12 hours of the sample. Bracketing CCVs are not required.

Analyze a CCV (AR1660) at least every 20 field samples for TO4A/TO10A. The laboratory does not perform a CCV for the remaining Aroclors unless requested for the project or by regulatory requirement.

A CCV (AR1660) must be analyzed at least every 12 hours of operation.

Aliquot 100uL of the needed standard into a vial. Add 4uL of the IS solution, and crimp tightly. . Inject the CCV standard onto the instrument in the same manner as performed for the initial calibration standards.

The data system calculates an average calibration factor (CF) for each set of 5 quantitation peaks. The percent difference from the average calibration factor in the initial calibration is then calculated.

For 8082A, the percent difference or drift must be within ±20% for the average of the quantitation peaks and the retention time (RT) must be within the established RT window. Acceptance criteria must be met on both columns.

For TO4A/TO10A, the percent difference or drift must be within ±15% for the average of the quantitation peaks and the retention time (RT) must be within the established RT window. Acceptance criteria must be met on both columns.

The AR1660 CCV standard must meet a resolution criteria of <75% for the triplet near the end of the AR1260 pattern on at least one column. The resolution may be calculated per the equation in Appendix C.

If the CCV fails, it may be repeated once. If repeat analysis fails, corrective action must be taken. If the two CCVs do not meet the criteria, recalibration is required prior to running samples.

10.3.4 Retention Time Updates

For 8082A analysis, all target analytes must have at least one CCV per 24 hours to update the retention time. If the samples have been screened and the Aroclors present in the samples are known, only those Aroclors must have the retention times updated daily.

For TO4A and TO10A analysis, only Aroclor 1660 is required to be analyzed every 24 hours. Update retention times of the other Aroclors as necessary.

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10.4 Troubleshooting

Check the following items in case of calibration failures:

- ICAL Failure – Perform injection port maintenance, install new guard column, check detector ends to see if detector jet has slipped. In extreme cases, install new columns, particularly if the chromatography has degraded as evidenced by peak shapes.
- CCV Failure – Perform Injection port maintenance; if injection port maintenance does not result in acceptable performance install a new guard column and remove one or more loops from each analytical column.
- Needle crushed during injection - Replace the needle and check the injection port for obstructions and check the autosampler for misalignment. Remove and clean the needle guide if necessary.
- Auto-sampler failure - Reset the auto-sampler and GC system. Replace the z-axis belt or clean the needle guide as necessary.
- Power failure - Reset run in ChemStation and re-acquire or re-initiate run sequence.

10.5 Sample Analysis

It is recommended that samples are screened to determine dilutions, Aroclor identifications, and the potential need for further sample cleanup prior to analysis.

Samples are typically screened at a 10x dilution against a single-point calibration curve. Screen data is not uploaded to TALS and is not reported.

10.6 Sample Analysis

Remove the extract from refrigerated storage and warm to room temperature.

Transfer 100 µL of extract to an autosampler vial. Add 4uL of IS solution, and crimp the vial tightly.

Place the vials in the autosampler in a sequence that begins with the calibration standards followed by the analysis of an ICV, QC samples, field samples and continuing calibration verification standards (CCVs).

Enter the sample ID's into the data acquisition program in the order that the samples were placed in the autosampler tray and initiate the analytical sequence.

Example analytical sequences follow:

Full Calibration Sequence	
Injection Number	**Lab Description**
1-2	Conditioning Blank
3-7	AR1221/1254 Calibration Block
8-12	AR1232/1262 Calibration Block
13-17	AR1242/1268 Calibration Block
18-22	AR1248 Calibration Block

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23-27	AR1660 and Surrogate Calibration Block
28	Initial Calibration Verification

Example Sample Analysis Sequence	
Injection Number	**Lab Description**
1-2	Conditioning Blank
3	Retention Time Update Standard(s)
4	AR1660 CCV
	CCVs for other Aroclors, as needed
5-24	Sample injections
	Repeat sample/CCV blocks as necessary

Cleaning blanks (PIBLK) consisting of hexane may be analyzed after high-level samples at the discretion of the analyst for samples which contain large concentrations of late-eluting compounds.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

The data processing system identifies the target analytes by comparing the retention time of the peaks to the established retention time windows.

Review and accept or reject the qualitative identifications made by the data processing system using the following guidelines:

Compare the retention time of the peak to the established RT window, taking into account the shift of the surrogate peaks. If the surrogate peaks have shifted, open the retention time window in the direction of the shift. The processing system identifies the peak in the retention time window that is closest to the expected retention time set in the Chrom method, so the peak may need to be re-identified if a shift has occurred. The data system does not recognize Aroclor patterns. The analyst manually identifies Aroclors by comparing the pattern in the samples to the patterns in the initial calibration standards. Weathering of PCB's in the environment may alter the PCB's to the point that the pattern no longer matches the pattern established for that Aroclor in the initial calibration. The laboratory takes the best pattern match approach to the identification and quantification of weathered PCB's.

Look for shoulders on the side of large peaks that may be peaks of interest. The processing system does not always automatically integrate shoulders from larger peaks, so manual integration (split) of the shoulder may be necessary.

Each target analyte must be detected on both columns for qualitative identification to be made.

11.2 Quantitative Identification

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Using an average of the chosen quantification peaks per Aroclor the data system calculates the corrected concentration for each target analyte using the equations given in Appendix C. If sample interference is suspected, the laboratory may remove quantification peaks per column, keeping a minimum of 3 quantitation peaks per Aroclor on each column. The lower value between the two columns is reported as the primary result, unless the project specifies otherwise and the TALS formatter uses another formatting rule.

11.3 Calculations

See Appendix C.

11.4 Data Review

See laboratory SOP BR-QA-019 for detailed data review requirements.

11.4.1 Primary Review

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Confirm qualitative and quantitative identification criteria using the criteria provided in Sections 11.1 and 11.2. If the data system does not properly integrate the peaks, perform manual integration in accordance with corporate SOP CA-Q-S-002.

Upload the data files from the data processing system (Chrom) to the laboratory information management system (TALS). Complete the batch information for standards and reagents and verify ICAL and QC sample associations. Review the results and set results to primary, secondary, acceptable or rejected as appropriate. Dilute and reanalyze samples whose results exceed the calibration range. The dilution analysis should result in a determination within the calibration range, preferably in the upper half of the calibration range. A more concentrated analysis is not necessary unless the project requires it. Dilution analyses may be performed to minimize matrix interference.

If a sample was analyzed immediately following a high concentration sample, review the results of the sample for any sign of carryover. If carryover is suspected, reanalyze the sample.

Create a non-conformance memo (NCM) for any calibration, QC and sample data that is reported outside established acceptance criteria and/or schedule necessary corrective action. Set batch to 1st level review and complete the data review checklist.

11.4.2 Secondary Data Review

Verify quantitative and qualitative identification in the initial calibration standards and spot check such for ~15% of the remaining data in the batch.

If manual integrations were performed, review each integration to verify that the integration meets the requirements for manual integration as specified in corporate SOP CA-Q-S-002. If an error is suspected or found consult with the analyst that performed the integration and request correction or notify the Department Supervisor, Operations Manager, or QA Manager. Do not “fix” the

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integration. Reintegration by a secondary data reviewer must not be performed except in limited circumstances as approved by the department supervisor or other laboratory management. In those instances where the secondary reviewer performs the integration, this person is now considered the primary analyst and each integration performed by the secondary reviewer must be subsequently reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified CA-Q-S-002.

Review project documents to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Verify that the acceptance criteria for the calibration and QC items listed in Table 1 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If not, initiate corrective actions and/or verify an NCM was created to document the criteria exception. Verify analytical results are qualified accordingly. Set batch to 2nd level review and complete the data review checklists.

Run the QC checker and fix any problems found. Run and review the deliverable. Fix any problems found. When complete set the method chain to lab complete and forward any paperwork to report/project management.

12.0 Method Performance

12.1 Method Detection Limit (MDL), Limit of Detection (LOD), and Limit of Quantitation (LOQ)

Establish a LOD and LOQ at initial method set up following the procedures specified in laboratory SOP BR-QA-005. Verify the LOD and LOQ at the frequency established for the method using the procedures specified in same SOP. The frequency of LOD and LOQ verification depends on the strictest frequency of the regulatory program for which the method supports. The frequency requirement is documented in a spreadsheet maintained by the QA Department.

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when there is a significant change in instrumentation or procedure.

Each analyst that performs the analytical procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory's quality system manual (QAM) and in the laboratory SOP for employee training.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of the SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

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13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001. The following waste streams are produced when this method is carried out.

- Vials containing sample extracts: Satellite container: 15 gallon bucket connected to a fume hood.
- Solvent Waste: Satellite container: 1 L glass bottle located in fume hood.

15.0 References / Cross-References

- SW-846 Method 8082A Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, February 2007.
- Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999 (EPA/625/R-96/010b)
- · Compendium Method TO-10A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), January 1999 (EPA/625/R-96/010b)
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-QA-002, Traceability, Handling, & Storage of Standards & Reagents
- Laboratory SOP BR-QA-011, Employee Training and Analyst Demonstration of Proficiency
- Laboratory SOP BR-EH-001, Tracking and Collection of Hazardous Waste
- Laboratory SOP BR-QA-014, Laboratory Records
- Laboratory SOP BR-QA-005, Procedures for Detection Limits, Limits of Detection, Limits of Quantitation
- Corporate SOP CA-Q-S-002, Manual Integrations
- Corporate SOP CA-Q-P-003, Calibration Curves and the Selection of Calibration Points
- Laboratory SOP BR-QA-005

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16.0 Method Modifications

Method Reference	Modification
TO4A 13.1/TO10A 12.1	The laboratory uses five-point calibration curves for all Aroclors instead of the three-point curves required in the reference method
	The laboratory calibrates with all Aroclors, not only 1242 and 1254 or 1260
	The use of internal standards has been incorporated with this method based on guidance from method 8082A section 11.6.8. Per that guidance, bracketing CCVs are not required.

17.0 Attachments

- Table 1: Target Compound List and Reporting Limit
- Table 1A: Accuracy and Precision Limits
- Table 2: Primary Materials Used
- Table 3: QC Summary & Recommended Corrective Action (8082A)
- Table 3: QC Summary & Recommended Corrective Action (TO4A & TO10A)
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables
- Appendix C: Equations

18.0 Revision History

BR-GC-005, Rev 13
This SOP is a complete rewrite of the procedure.

Previous Revisions are retained by the QA department.

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Table 1: Routine Target Analyte List & Reporting Limits (RL)

ANALYTE	Routine Reporting Limit (RL)[1,2]				
	Water (ug/L)	Solid (ug/Kg)	Air (ug/m^3 TO10A)[3]	Air (ug/m^3 TO4A)[4]	Air (ug/PUF)
AR1016	0.50	17	0.10	0.00033	0.10
AR1221	0.50	17	0.10	0.00033	0.10
AR1232	0.50	17	0.10	0.00033	0.10
AR1242	0.50	17	0.10	0.00033	0.10
AR1248	0.50	17	0.10	0.00033	0.10
AR1254	0.50	17	0.10	0.00033	0.10
AR1260	0.50	17	0.10	0.00033	0.10
AR1262	0.50	17	0.10	0.00033	0.10
AR1268	0.50	17	0.10	0.00033	0.10

[1]The routine RL is the unadjusted value that can be achieved in a blank matrix.
[2]The RL for tissue matrix is project defined.
[3]RL based on a collection volume of 1m^3 or 1,000L
[4]RL based on a collection volume of 300m^3 or 300,000L

Table 1A: Routine Accuracy and Precision Limits[1]

Analyte	In-House Limits (%R)			Precision (RPD) (≤)
	Water	Solid	Air	
AR1016	55-120	55-120	65-125	30
AR1260	60-125	55-125	65-125	30
Surrogate: Decachlorobiphenyl (DCB)	30-150	45-125	60-120	NA
Surrogate:TCX (Advisory)[2]	55-120	30-130	60-120	NA

[1] The limits in this table are those used as of the effective date of this SOP. Current limits are stored in the LIMS database.
[2] The control limits for TCX (Tetrachloro-m-xylene) are advisory. Corrective action is not performed when recovery is outside limits.

Table 2: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

[1] Always add acid to water to prevent violent reactions.
[2] Exposure limit refers to the OSHA regulatory exposure limit.

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Table 3: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action (8082A)

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	RSD for each analyte ≤ 20% -OR- Linear Regression: r ≥ 0.995	Correct problem, reanalyze, and repeat calibration.
ICV	After each initial calibration	(% R) ± 20% from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 20 field samples or 12 hours (whichever is sooner).	% Difference or Drift ≥20%	See Section 10.3. Perform maintenance such as Injection Port maintenance, check for leaks, and change the lines / septa.
MB	One per extraction batch of 20 or fewer field samples	Target Analyte < RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or non-conformance memo (NCM). If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
LCS	One per extraction batch of 20 or fewer field samples	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance memo (NCM). Flag all reported values outside of control limits
LCSD	One per extraction batch of 20 or field fewer samples, when MS/MSD is not present	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance memo (NCM). Flag all reported values outside of control limits.
MS/MSD	One per extraction batch of 20 or fewer field samples	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
SD	Per client request	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Internal Standard	All field and QC samples	Area between 50-200% of area of daily calibration internal standard area	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate	All field and QC samples	See Table 1A	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1] *The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

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Table 3A: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action (EPA TO4 & TO10)

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Before sample analysis, when CCVs indicate calibration is no longer valid; after major instrument maintenance	Option 1: RSD for each analyte ≤ 20%	Correct problem, reanalyze, repeat calibration.
ICV	After each initial calibration	(% R) ± 15 % from expected value	Correct problem and verify second source standard. If that fails, repeat initial calibration.
CCV	Daily before sample analysis, every 20 field samples or 12 hours (whichever is sooner).	% Difference or Drift ±15%	Re-analyze once, if still outside criteria perform corrective action, sequence can be re-started if two successive CCVs pass, otherwise repeat ICAL and all associated samples since last successful CCV, unless CCV is high and bracketed samples are non-detects.
Method Blank	One per extraction batch of 20 or fewer field samples	Target Analyte < RL	Examine project DQO's and take appropriate corrective action, which may include re-analysis of MB, re-extraction of batch, and/or NCM. Corrective action must be documented in an NCM. If there are no detects in samples, or if all detects are > 10 X MB level, re-prep and reanalysis may not be required.
Field Blank	One per sampling event	Target Analyte < RL	Notify client.
LCS	One per extraction batch of 20 or fewer field samples	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance memo (NCM). Flag all reported values outside of control limits
LCSD	One per extraction batch of 20 or fewer field samples, when MS/MSD is not present	See Table 1A	Examine project DQO's and take appropriate corrective action, which may include re-analysis of LCS, re-extraction of batch, and/or non-conformance memo (NCM). Flag all reported values outside of control limits.
Field Spike	One per sampling event	%R (65-125)	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract.
Internal Standard	All field and QC samples	Area between 50-200% of area of daily calibration internal standard area	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze and/or re-extract. Flag all reported values outside of control limits.
Surrogate	All field and QC samples	%R (60-120)	Evaluate data and determine if a matrix effect or analytical error is indicated. If analytical error, re-analyze or re-extract. If matrix effect, review project DQOs to determine if a matrix effect must be confirmed by re-analysis. Flag all reported values outside of control limits.

[1]*The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.*

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Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: 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 which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: 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 the standards.

Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Data Qualifier: a letter designation or symbol appended to an analytical result used to convey information to the data user. (Laboratory)

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

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Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are only produced in this range and qualified with the proper data reporting flag when a project requires this type of data reporting.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

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Appendix B: Standard Preparation Tables

The standard formulations contained in this Appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in hexane using Class A volumetric glassware and Hamilton syringes. Unless otherwise specified for a standard solution, assign an expiration date of 1 year from date of preparation unless the parent standard expires sooner in which case use the earliest expiration date. Store the prepared solutions under refrigeration at a temperature of 4ºC (±2) and protect from light. See laboratory SOP BR-QA-002 *Traceability, Handling, & Storage of Standards & Reagents* for further guidance.

Intermediate Calibration Standards (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
AR1660[1]	Restek #32039	Aroclor 1016 Aroclor 1260	1000	0.40	40	10
AR1254	Restek #32011	Aroclor 1254	1000	0.40	40	10
AR1248	Restek #32010	Aroclor 1248	1000	0.40	40	10
AR1242	Restek #32009	Aroclor 1242	1000	0.40	40	10
AR1232	Restek #32008	Aroclor 1232	1000	0.40	40	10
AR1221	Restek #32007	Aroclor 1221	1000	0.40	40	10
AR1262	Restek #32409	Aroclor 1262	1000	0.40	40	10
AR1268	Restek #32410	Aroclor 1268	1000	0.40	40	10

[1] *Standard is a mix of AR1016/AR1260. Concentration shown is the concentration of each Aroclor in the mixed standard.*

Intermediate ICV Standard (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
AR1660	Restek	Aroclor 1016 Aroclor 1260	1000	0.40	40	10

Surrogate Solution (10 mg/L)

Parent Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Pesticide Surrogate	Restek #3200	TCX DCB	1000	0.40	40	10

Working ICV Standard (200 ug/L)

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
Intermediate ICV	Laboratory Prepared	Aroclor 1016 Aroclor 1260	10	0.80	40	200
Surrogate	Laboratory Prepared	TCX DCB	10	0.080		20

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AR1660 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1660 Intermediate	Laboratory Prepared	Aroclor 1016 Aroclor 1260	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1660 calibration standards*

AR1660 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1660 Level 5	AR1660 CAL Level 4	800	20	40	400
AR1660 Level 5	AR1660 CAL Level 3	800	10	40	200
AR1660 Level 5	AR1660 CAL Level 2	800	5.0	40	100
AR1660 Level 5	AR1660 CAL Level 1	800	2.5	40	50

AR1221 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1221 Intermediate	Laboratory Prepared	Aroclor 1221	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1221 calibration standards*

AR1221 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1221 Level 5	AR1221 CAL Level 4	800	20	40	400
AR1221 Level 5	AR1221 CAL Level 3	800	10	40	200
AR1221 Level 5	AR1221 CAL Level 2	800	5.0	40	100
AR1221 Level 5	AR1221 CAL Level 1	800	2.5	40	50

AR1232 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1232 Intermediate	Laboratory Prepared	Aroclor 1232	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

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[1] *This standard is the parent standard for each level of the AR1232 calibration standards*

AR1232 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1232 Level 5	AR1232 CAL Level 4	800	20	40	400
AR1232 Level 5	AR1232 CAL Level 3	800	10	40	200
AR1232 Level 5	AR1232 CAL Level 2	800	5.0	40	100
AR1232 Level 5	AR1232 CAL Level 1	800	2.5	40	50

AR1242 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1242 Intermediate	Laboratory Prepared	Aroclor 1242	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1242 calibration standards*

AR1242 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1242 Level 5	AR1242 CAL Level 4	800	20	40	400
AR1242 Level 5	AR1242 CAL Level 3	800	10	40	200
AR1242 Level 5	AR1242 CAL Level 2	800	5.0	40	100
AR1242 Level 5	AR1242 CAL Level 1	800	2.5	40	50

AR1248 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1248 Intermediate	Laboratory Prepared	Aroclor 1248	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1248 calibration standards*

AR1248 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1248 Level 5	AR1248 CAL Level 4	800	20	40	400
AR1248 Level 5	AR1248 CAL Level 3	800	10	40	200
AR1248 Level 5	AR1248 CAL Level 2	800	5.0	40	100
AR1248 Level 5	AR1248 CAL Level 1	800	2.5	40	50

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AR1254 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1254 Intermediate	Laboratory Prepared	Aroclor 1254	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1254 calibration standards*

AR1254 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1254 Level 5	AR1254 CAL Level 4	800	20	40	400
AR1254 Level 5	AR1254 CAL Level 3	800	10	40	200
AR1254 Level 5	AR1254 CAL Level 2	800	5.0	40	100
AR1254 Level 5	AR1254 CAL Level 1	800	2.5	40	50

AR1262 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1262 Intermediate	Laboratory Prepared	Aroclor 1262	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard for each level of the AR1262 calibration standards*

AR1262 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1262 Level 5	AR1262 CAL Level 4	800	20	40	400
AR1262 Level 5	AR1262 CAL Level 3	800	10	40	200
AR1262 Level 5	AR1262 CAL Level 2	800	5.0	40	100
AR1262 Level 5	AR1262 CAL Level 1	800	2.5	40	50

AR1268 Calibration Standard: CAL Level 5 (800 ug/L)[1]

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1268 Intermediate	Laboratory Prepared	Aroclor 1268	10	8.0	100	800
Surrogate	Laboratory Prepared	TCX DCB	10	0.80		80

[1] *This standard is the parent standard fo each level of the AR1268 calibration standards*

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AR1268 Calibration Standard(s): CAL Levels 1- 4

Parent Standard	Calibration Standard	Parent Standard Concentration (ug/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (ug/L)
AR1268 Level 5	AR1268 CAL Level 4	800	20	40	400
AR1268 Level 5	AR1268 CAL Level 3	800	10	40	200
AR1268 Level 5	AR1268 CAL Level 2	800	5.0	40	100
AR1268 Level 5	AR1268 CAL Level 1	800	2.5	40	50

Internal Standard Solution (500 ug/L)

Parent Standard	Vendor	Component	Parent Standard Concentration (mg/L)	Volume Added (uL)	Final Volume (mL)	Final Concentration (ug/L)
1-Bromo-2-nitrobenzene Stock	Restek p/n 32279	1-Bromo-2-nitrobenzene	1000	20	40	500

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Appendix C: Equations

Relative Response Factor Calculation

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
AX =Peak height for the Aroclor peak to be measured.
Ais = Peak height for specified internal standard.
Cis = Amount of the internal standard injected (ng).
Cx = Amount of the target compound or DMC injected (ng).

Mean Relative Response Factor Calculation

$$\overline{RRF} = \frac{\sum_{i=1}^{n} RRF_i}{n}$$

Where:
n = The number of calibration standards

Percent Relative Standard Deviation Calculation

$$\%RSD = \frac{Standard\ Deviation}{Mean} \times 100$$

$$Standard\ Deviation = \sqrt{\frac{\sum_{i=1}^{n} \left(x_i - \overline{x}\right)^2}{n-1}}$$

Where:
xi = Each individual value used to calculate the mean
$\overline{x}$ = The mean of n values
n = The total number of values

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Relative Response Factor Percent Difference Calculation

$$\%Difference_{RRF} = \frac{RRF_C - \overline{RRF_i}}{\overline{RRFi}} x100$$

Where:

$\overline{RRF_i}$ = Mean Relative Response Factor from the most recent initial calibration meeting
technical acceptance criteria
RRFc = Relative Response Factor from CCV standard.

Percent Drift = Calculated Concentration – Theoretical Concentration X 100%
Theoretical Concentration

Percent Recovery

$$\%R = \frac{C_s}{C_n} \times 100\%$$

Cs = Concentration of the Spiked Field or QC Sample
Cn = Nominal Concentration of Spike Added

Percent Recovery (%R) for MS/MSD

$$\%R = \frac{C_s - C_u}{C_n} \times 100\%$$

Cs = Concentration of the Spiked Sample
Cu = Concentration of the Unspiked Sample
Cn = Nominal Concentration of Spike Added

Relative Percent Difference

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100\%$$

C1 = Measured Concentration of First Sample
C2 = Measured Concentration of Second Sample

Sample Concentration
Note: The concentrations of the 3-5 peaks chosen for quantification, determined by peak height, are calculated and the average is then taken for final calculation.

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Concentration of Water Samples

$$Concentration\ ug/L = \frac{(A_x)(I_s)(V_t)(DF)(GPC)}{(A_{is})(\overline{RRF})(V_o)(V_i)}$$

Where:
Ax = Peak height for the peak to be measured.
Ais = Peak height for the internal standard.
Is = Amount of internal standard injected in nanograms (ng).
Vo = Volume of water extracted in milliliters (mL).
Vi = Volume of extract injected in microliters (µL).
Vt = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)
GPC = GPC Factor (If no GPC cleanup is performed, GPC = 1.0)

Concentration of Soil and Sediment Samples

$$Concentration\ ug/Kg\ (Dry\ Weight\ Basis) = \frac{(A_x)(I_s)(V_t)(DF)(GPC)}{(A_{is})(\overline{RRF})(V_i)(W_s)(D)}$$

Where:
Ax = Peak height for the peak to be measured.
Ais = Peak height for the internal standard.
Is = Amount of internal standard injected in nanograms (ng).
Ws = Weight of sample extracted in grams (g)
Vi = Volume of extract injected in microliters (µL).
Vt = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)
D = $\frac{100 - \%Moisture}{100}$
GPC = GPC Factor (If no GPC cleanup is performed, GPC = 1.0)

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Concentration of Air Samples (ug/PUF)

$$\text{Concentration ug/PUF} = \frac{(A_x)(I_s)(1PUF)(DF)}{(A_{is})(\overline{RRF})(V_o)(V_i)}$$

Where:
Ax = Peak height for the peak to be measured.
Ais = Peak height for the internal standard.
Is = Amount of internal standard injected in nanograms (ng).
Vo = Volume of water extracted in milliliters (mL).
Vi = Volume of extract injected in microliters (µL).
Vt = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)

Concentration of Air Samples (ug/m3)

$$\text{Concentration ug/m3} = \frac{(A_x)(I_s)(V_t)(DF)}{(A_{is})(\overline{RRF})(V_o)(V_i)}$$

Where:
Ax = Peak height for the peak to be measured.
Ais = Peak height for the internal standard.
Is = Amount of internal standard injected in nanograms (ng).
Vo = Volume of air sampled via PUF apparatus (m3).
Vi = Volume of extract injected in microliters (µL).
Vt = Volume of the concentrated extract in microliters (µL)
$\overline{RRF}$ = Mean Relative response factor determined from the initial calibration standard.
DF = Dilution Factor (If no dilution is performed, DF = 1.0)

Resolution

(Height of Valley / Average of the Two Peak Heights)*100%

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BR-EX-025

Media Prep and Extraction Procedure for PCBs in Ambient Air by SW‑846 3540 and 3541 (EPA TO4/ TO10)
(Rev. 2.1, 08/28/18)

Title: Media Prep and Extraction Procedure for PCBs in Ambient Air by SW-846 3540 and 3541 (EPA TO4/TO10)

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1.0 Scope and Application

This standard operating procedure (SOP) describes the laboratory procedure for the preparation of air sampling media and the extraction of ambient air samples collected on polyurethane foam (PUF).

1.1 Analytes, Matrix(s), and Reporting Limits

Refer to the SOP for the determinative method for analyte lists and reporting limits.

2.0 Summary of Method

Target analytes are extracted from a sorbent cartridge with 10% diethyl ether in hexane using soxhlet extraction. If necessary, the extract is dried with anyhydrous sodium sulfate and concentrated to 10 mL then relinquished to the analytical department for analysis by GC/ECD.

This SOP is based on the procedures given in the following reference method:

- Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD). USEPA Office of Research and Development; January 1999.
- Compendium Method TO10A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD). USEPA Office of Research and Development; January 1999.
- This SOP is based on SW846 Method 3540C “Soxhlet Extraction”, Revision 3, December 1996.

If the laboratory’s procedure includes modifications from the above reference method(s), a list of modifications will be provided in Section 16.0.

3.0 Definitions

A list of terms and definitions are provided in Appendix A.

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing equipment that can cause interference and/or elevated baselines in chromatography. All reagents and solvents used during this procedure should be reagent grade or high purity in order to minimize interference. All glassware must be cleaned in accordance with laboratory SOP BR-EX-017 and rinsed with acetone and methylene chloride prior to use.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

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5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

Diethyl ether is a flammable and peroxide-forming compound. Peroxides are highly reactive and/or explosive compounds. Diethyl ether may form peroxides when stored or evaporated to dryness. Once a peroxide has been formed, it can be detonated by simply moving the container. **Never** attempt to move a container when the shelf life has been exceeded, a container has been opened for more than 6 months, or there is evidence of peroxide formation. Visible evidence of potential peroxide formation includes crystals around the cap, a viscous layer at the bottom of the container or rust around the surface of the can. Contact your Environmental Health and Safety Coordinator or Department Manager for instructions.

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. **NOTE: This list does not include all materials used in the method.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

6.0 Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment used meets the specification of this SOP.

6.1 Extraction Equipment

- Circular, heated water bath equipped with chilled water condensers. Capacity to hold eight Soxhlet extractors and maintain temperature control at 90°C (±5°C). Organomation Model Number 130 or equivalent.
- Soxhlet Extractor: 55/50 Top Joint. AMK Catalog Number EX-03B or equivalent.
- 500 mL flat bottom flask. AMK Catalog Number RB1-500 or equivalent.
- Teflon Ultra Pure PTFE Boiling Stones. Chemware Catalog Number 0919120 or equivalent.

6.2 Extract Concentration (KD Apparatus)

- Concentrator tube, 10 mL graduated. ChemGlass Catalog Number CG-1316-11 or equivalent
- Snyder Column, Three ball macro AMK Catalog Number SC2-01 or equivalent
- Snyder Column, Two ball micro AMK Catalog Number SC3-01 or equivalent
- Evaporation Flask, 500 mL attached to concentrator tube with clip. AMK Catalog Number KDF-500 or equivalent
- Filter Funnels: 100mm diameter for filtration/drying, Fisher Scientific or equivalent.
- Boiling Chips, silicon carbide, approximately 10/40 mesh, solvent extracted in methylene chloride. Troemner Catalog Number 133B or equivalent.

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- Heating mantle rheostat controlled for water bath capable of temperature control (± 5°C). ChemGlass Catalog Number PL3122 or equivalent.
- Water Bath, capable of temperature control to ±5°C. Barnstead Corporation Catalog Number HM0500-HS1 or equivalent.
- Solvent Vapor Recovery System, Kontes K-54000-1006, K-547300-000, Ace Glass Catalog Number 6614-30 or equivalent.
- FMS Power-Vap Concentrator System ™ capable of heating sample to 70C under Nitrogen gas flow, or equivalent

6.3 Sampling Media

- Glass Cartridge w/support screen Shawnee Catalog Number PS1-4 or equivalent.
- 3" Polyurethane Foam (PUF) Plugs; Shawnee or equivalent.
- QMA-4" Quartz Filter, 102-millimeter bindless quartz microfiber filter, Whatman or equivalent.
- Sorbent Tube, PUF SKC Catalog Number 226-92 or equivalent (Use for TO10A)

6.4 Miscellaneous

- Disposable glass Pasteur pipette and bulb.
- Teflon tape
- 0.5 mL – 2.0 mL Hamilton Gastight® syringes or equivalent.
- Aluminum Foil
- Pressure Cooker with vacuum & carbon cartridges.
- 16 mL Clear vial with a Teflon lined Cap, Fisher or equivalent
- Glass wool

7.0 Reagents and Standards

7.1 Reagents

- Sodium Sulfate (granular, anhydrous), Na_2SO_4. J.T. Baker or equivalent. Purify by heating at 400°C for at least 4 hours
- Methylene Chloride (CH_2C_{12}), Pesticide quality, J.T Baker or equivalent.
- Hexane, (C_6H_{14}), Pesticide Quality. J.T. Baker or equivalent.
- Acetone, ($(CH_3)_2CO$), Pesticide quality. J.T. Baker or equivalent.
- Anhydrous Diethyl Ether $(C_2H_5)_2O$; JT Baker or equivalent

7.2 Prepared Reagents

10% Ether/Hexane: Mix 1800 mL of hexane with 200 mL of anhydrous diethyl ether. Store in a 4L amber glass bottle at room temperature.
10% Ether/Acetone: Mix 1800 mL of Acetone with 200 mL of anhydrous diethyl ether. Store in a 4L amber glass bottle at room temperature.

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7.3 Standards

Purchase stock standards as certified solutions from commercial vendors. Prepare surrogate and spiking solutions in the laboratory by diluting a known volume of the stock standard solutions in an appropriate solvent. Record the preparation of standard in the LIMS (TALS) module established for this purpose.

Store prepared standard solutions in glass containers at 4°C or below. Unless otherwise specified, assign an expiration date of 6 months from the date of preparation or in accordance with the expiration date of the parent standard, whichever is sooner.

8.0 Sample Collection, Preservation, Shipment and Storage

Procedures for the preparation and certification of sampling cartridges are provided in Section 10.1.

The laboratory does not perform sample collection so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method. Note the reference method includes requirements for a field blank and field spike per sampling event and provisions for collection of these QC samples should be included in the client's sampling plan.

Listed below are laboratory recommended minimum sample size, preservation and holding time specifications:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Air	PUF Cartridge	1 per sample	0-6°C	7 days	EPA TO-4A and EPA TO10

[1]The holding time for extraction is determined from the date of sample collection.

NOTE: Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after release of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See Analytical SOP
Field Blank (FB)	With each sampling event	See Analytical SOP
Field Spike (MS)	With each sampling event	See Analytical SOP

- A Method Blank (MB) is a blank PUF plug processed with 20 or fewer samples but is not shipped to the field.
- A Field Blank (FB) is a PUF cartridge that is prepared in the laboratory placed in a sealed container shipped to the field and returned to the laboratory without drawing air through the sampler with other project samples. The field spike is the method specified QC check for recovery of target analytes.
- A Field Spike (MS) is a PUF cartridge prepared by the laboratory and spiked with a known amount of standard solution by the laboratory. The PUF cartridge is placed in a sealed container and shipped to the field and returned to the laboratory without drawing air through the sampler with other project samples.

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10.0 Procedure

10.1 Preparation and Certification of PUF Sampling Cartridge (EPA TO4A)

This section describes the preparation and certification of the PUF sampling cartridge for TO4A. For sampling media by EPA TO10 the laboratory purchases sorbent tubes and PUF pre-certified from the vendor, therefore procedures for preparation and certification of PUF for TO10 are not included in this SOP.

10.1.1 Bake the quartz filters in an oven maintained at a temperature of 400°C for 5 hours. Wrap the clean filters in aluminum foil and set aside for shipment to the client or for later combination with the PUF glass assembly cartridge. Document the logbook designated for this purpose.

10.1.2 Turn on water baths for Soxhlet extractor at least 30 minutes prior to beginning procedure to allow to warm up.

10.1.3 Place the PUF plug into a soxhlet extractor and extract with 10% Acetone/Ether for 16 hours at approximately 4-6 cycles per hour. Remove the plug using stainless steel forceps that have been pre-rinsed with acetone. Place the plugs in a pressure cooker and place the cover on the pressure cooker. Attach the vacuum hose and turn the unit on, start vacuum and dry the plugs overnight or until no solvent odor is detected.

10.1.4 Prepare a glass cartridge for each sample by placing a stainless steel screen in the sampling cartridge then rinse the cartridge with hexane.

Put on nitrile gloves and rinse the stainless steel forceps with acetone. Use the forceps to remove a plug from the pressure cooker and place the plug into a glass sampling cartridge. Wrap the cartridge with aluminum foil and place the cartridge in a Ziploc bag. Repeat for each plug.

10.1.5 For each lot of filters and cartridges prepared, extract 1 filter and 1 PUF cartridge by soxhlet extraction, concentrate using KD technique to 1 mL and relinquish to the analytical department for analysis. DO NOT add any spiking solutions. Identify this as “MDAB” in the prep batch. When the lots are certified clean, place the cartridges in inventory or release to sample management for shipment to the field.

10.2 Field Spike

If a field spike is requested for the job, add the proper volume of surrogate spike solution to the prepared sampling cartridges prior to shipment to the customer. Refer to the extraction conditions workbook for type and volume of surrogate spike to add.

10.3 Extraction

10.3.1 Prepare the extraction glassware using the procedures described in laboratory SOP BR-EX-017 then rinse with 10% diethyl ether in hexane.

Samples may be extracted by soxtherm (SW846 3541, see sop BR-EX-027) or by soxhlet (SW846 3540). The extraction method to use will be specified in the TALS method chain.

10.3.2 For extraction using Soxtherm (SW-846 3541), use approximately 120mLs of10% diethyl ether in hexane as the extraction solvent.

- Add the appropriate spiking solution (see Extractions Conditions Workbook).
- Follow the Extraction section in SOP BR-EX-027 for these samples.

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10.3.3 For extracting using Soxhlet (SW-846 3540), turn on water baths for Soxhlet extractor at least 30 minutes prior to beginning procedure to allow to warm up.

- Place the PUF plug into a soxhlet extractor using stainless steel forceps that have been pre-rinsed with acetone.
- Add approximately 300mLs 10% ether/hexane to the Soxhlet extractor.
- Add the appropriate volume of spiking solution (see Extractions Conditions Workbook).
- Add an additional ~100mLs of 10% ether/hexane to the apparatus.
- Place assembly on hot water bath, attach condensers, and allow to extract for at least 16 hours (must cycle 4-6 times per hour; 10-15 minutes per cycle).
- Once extracted (after 16 hours), filter through sodium sulfate to remove any water (if necessary). Once complete, move on to the clean-up and concentration.

  NOTE: If water is observed, NCM as an observation.

10.3.4 Follow the appropriate clean-up method (See SOP BR-EX-002) and concentrate using the Power-VAP systems (see SOP BR-EX-031).

10.3.5 Refer to the Extraction Conditions Workbook, located in the SOPs_Controlled_Distribution folder, for spike information and final extract volumes.

11.0 Calculations / Data Reduction

11.1 Calculations

There are no calculations applicable to this procedure.

11.2 Data Review

11.2.1 Primary Review

Create a TALS batch and enter the sample information and complete the batch editor and worksheets. Review your work to ensure the extraction is consistent with the procedures given in this SOP and with any project specific requirements. Be advised that modification to the procedures, including initial and final extract volumes, is not permitted without documented management or project specific approval. Set the status of the batch to 1st level review.

11.2.2 Secondary Review

Review the TALS batch worksheets and batch editor to verify that the worksheets are complete and include all supporting information. Verify the procedure performed is consistent with this SOP and any modifications if performed are properly documented and were performed with approval. Verify that the sample amounts and initial and final volumes used are correct. Check the batch for the items listed in the data review checklist and set the status of the batch to second level review.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) Study must be determined for each test method associated with this extraction procedure during initial method set-up or prior to the analysis of field samples. The MDLs are verified annually or after major instrument maintenance. The procedure for the determination of MDLs is described in laboratory SOP BR-QA-005.

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12.2 Demonstration of Capabilities (DOC)

Each analyst must complete an Initial Demonstration of Capability prior to unsupervised performance of this method.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to laboratory SOP BR-EH-001.

15.0 References / Cross-References

- Compendium Method TO-4A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD). USEPA Office of Research and Development; January 1999.
- Compendium Method TO10A, Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD). USEPA Office of Research and Development; January 1999.
- Corporate Environmental Health and Safety Manual (CW-E-M-001)
- Laboratory SOP BR-QA-005
- Laboratory SOP BR-QA-019
- Laboratory SOP BR-EH-001
- Laboratory SOP BR-EX-002
- Laboratory SOP BR-EX-017
- Laboratory SOP BR-EX-027
- Laboratory SOP BR-EX-031

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16.0 Method Modifications

Modification Number	Method Reference	Modification
1	EPA TO-4A Section 10.2.6	PUFs are not wrapped in hexane-rinsed aluminum foil or placed in an aluminum shipping container sealed with Teflon tape. Laboratory experience indicates that the aluminum foil used by laboratory is clean and does not require the additional rinse; the laboratory stores the prepared PUF cartridges in Ziploc bags.
2	EPA TO-4A Section 12.1.2	The laboratory does not bake glassware in an oven at 500°C.
3	EPA TO-4A Section 12.1.5	The laboratory uses 400 mL of extraction solvent in a 500 mL soxhlet apparatus instead of 700 mL of extraction solvent in order to reduce the production of hazardous waste solvent.
4	EPA TO-4A	Lab uses nitrile gloves instead of polyester because they provide better protection from solvents.
5	EPA TO-10A	The laboratory uses 10% diethyl ether/ hexane as the extraction solvent instead of 5% diethyl ether/ hexane.
6	TO4A	The laboratory may extract samples by automated soxhlet (soxtherm) on a project specific basis.

17.0 Attachments

- Table 1: Primary Materials Used
- Appendix A: Terms and Definitions
- Appendix B: Standard Preparation Tables

18.0 Revision History

Revision 2.1: 08-21-2018
- Title Page: Updated signatures

Section 2.0: added reference to SW-846 3540
Section 6.2: added Power-VAP systems to equipment list
Section 7.2: added 10% Ether/Acetone to reagents
Section 8.0: updated temperature range from 4 to 0-6 for consistency
Section 10.1: replaced Acetone with 10%Ether/Acetone
Section 10.1: specified to not add spikes to cleaned PUF for verification analysis and specified QC type
Section 10.3: clarified procedure for 3541 and 3540.
Throughout: minor typographical clarifications and formatting corrections

Previous revisions are retained by the QA department.

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Table 1: Primary Materials Used

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of with six months.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

[1] *Always add acid to water to prevent violent reactions.*
[2] *Exposure limit refers to the OSHA regulatory exposure limit.*

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Appendix A: Terms and Definitions

Acceptance Criteria: specified limits placed on characteristics of an item, process or service defined in requirement documents.

Accuracy: 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 which are due to sampling and analytical operations; a data quality indicator.

Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).

Batch: environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.

Calibration: 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 the standards.

Calibration Standard: A substance or reference used to calibrate an instrument.

Continuing Calibration Verification (CCV): a single or multi-parameter calibration standard used to verify the stability of the method over time. Usually from the same source as the calibration curve.

Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.

Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Spike (MS): a field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a second replicate matrix spike

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Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Stock Standard: a solution made with one or more neat standards usually with a high concentration. Also known as a primary standard. Stock standards may be certified solutions purchased from a vendor.

Surrogate: a substance with properties that mimic the analyte of interest but that are unlikely to be found in environmental samples.

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Appendix B: Standard Formulation Tables

The standard formulations contained in this appendix are recommended and are subject to change. If the concentration of the stock standard is different than those noted in this table, adjust the standard preparation formulation accordingly. Unless otherwise specified, prepare the standard solutions in a Class A volumetric glassware using Hamilton syringes. Stock standards must be replaced after one year or sooner if QC tests indicate a problem. For working standards, unless otherwise specified, assign an expiration date of 6 months from date of preparation unless the stock standard expires sooner in which case the earliest expiration date is used. See laboratory SOP BR-QA-002 Standard & Reagent Preparation for further guidance. Standards should be stored in a refrigerator in PTFE-sealed containers.

Pesticide Surrogate Solution

Stock Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Pesticide Surrogate	Restek #3200	DCB	200	1.0	1000	0.20
		TCMX	200			0.20

Solvent: Acetone

8082 Field Spike Solution

Stock Standard	Vendor	Component	Stock Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
AR1016/1260 Mix	Restek #32039	AR1016	1000	1.0	200	5.0
		AR1260	1000			5.0

Solvent: Acetone

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Standard Operation Procedure #1: Sediment Sampling and Processing

The protocol set forth in this Standard Operating Procedure (SOP) outlines the required field procedures for the collection of sediment via vibracoring, manual core collection, and/or grab samples using a hand-held ponar dredge. Vibracoring techniques will be used as the primary collection method unless the target sampling location cannot be accessed; manual coring will be performed at locations with limited access. Grab samples via the ponar dredge will be collected in locations with a thin layer of sediment or if the material is not able to be recovered via coring. Details regarding the sampling objectives, design, and quality assurance requirements are provided in the Construction Quality Assurance Plan (CQAP) and Quality Assurance Project Plan (QAPP) Worksheets (Appendix C); the procedures outlined here will be performed in compliance with the CQAP and QAPP Worksheets.

A. Sediment Sampling Procedure Using Vibracoring and Core Processing

Materials:
The following materials will be available, as required, during sediment sampling via vibracoring and associated core processing:

- Health and safety and personal protective equipment (as required by the Health and Safety Plan [HASP], Contractor Safety Work Plan [CSWP], and associated Job Safety Analyses [JSAs])
- 24-foot aluminum decked coring boat equipped with outboard motor, automated anchoring system, derrick, and winch assembly
- Vibracoring device (Rossfelder P-3C) and associated equipment
- 3” aluminum tubing with end caps
- Fabricated core tube rack for secondary containment and for core icing
- Pipe cutter and electric shears (or other appropriate devices)
- Real-Time Kinematic (RTK) survey equipment
- Disposable aluminum pans
- Disposable spatulas
- Appropriate sample containers and forms
- Coolers with ice
- Field notebook and computer
- Camera
- Surveyor's rod
- Duct tape
- Calibrated probe rod
- Six-foot rule

Sediment Sampling Procedures
Sediment samples will be collected using a specially designated boat designed for vibracoring. Samples will be collected at designated locations. The procedures for collection of sediment samples via vibracoring are provided below.

A1. Don health and safety equipment (as required in the HASP, CSWP, and JSAs).

A2. Setup RTK survey equipment (includes master station and rover). Set master station on known control point and check coordinates. Set rover on different control point and check coordinates. Setups are considered acceptable if coordinates check to within 0.1 feet horizontally and vertically. If not within accuracy, perform

troubleshooting efforts including verifying control point coordinates and numbers, confirm no physical impacts to control points, etc. If accuracy cannot be achieved, utilize different control point(s).

A3. Setup rover station on the sampling vessel as the rover station will be used to navigate to the sample location. The rover station will be placed within a specially designed holder situated on the center point of the moon pool cover. The distance from the deck to the water is a known measurement for determining the water surface elevation.

A4. Use RTK surveying techniques to maneuver the sampling vessel to the target sample location. Secure the vessel in place using steel spud anchoring system.

A5. Measure the total depth of water using a surveyor's rod to the nearest 0.1 foot and document, and survey the water surface elevation using RTK survey equipment.

A6. Obtain the sediment depth through probing by manually pushing a 5/8-inch outside diameter calibrated steel pipe into the sediment as far as possible using reasonable human force 3 to 5 feet away from the target location. The depth of refusal will be interpreted as the interface between soft sediment and rock or stiff bottom. Record sediment depth, type, and presence of debris or obstructions.

- If probing indicates little to no sediment present, expand the probing efforts to cover an area around the sampling boat while staying within the removal area boundary (approximate 20 feet) in an effort to identify a location with sediment. If no sediment can be identified based on probing efforts, abandon the location and document probing attempts.

A7. Once the targeted area is deemed suitable for core collection, select an appropriate length of 3-inch outside diameter aluminum core tube based on the probing information. Deeper sediments will be sampled with core tubes custom cut on the boat from 12 foot tube sections.

A8. Secure the selected coring tube into the vibracoring head.

A9. Lower the coring apparatus vertically through the water column until the core tube just reaches the top of sediment then vibrate the core to refusal (approximately 1 minute with no further vertical movement into consolidated sediment or encountering the top of rock). Measure and record the depth of core tube penetration into the sediments.

A10. Pull the coring apparatus upward out of the river bottom using the recovery winch, and raise it to the surface while maintaining the core in a vertical position.

A11. 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 sample vessel. Secure the cap in place with duct tape when brought on board the vessel. Note that with consolidated sediment (clays) the core end may be capped after it breaks the water surface.

A12. Check for core acceptability by observing the overlying water for significant turbidity or if sediment is observed at the top of the core tube indicating over penetration (i.e., core tube was too short). Unacceptable cores will be discarded and recollected.

A13. If acceptable, estimate and record the recovered length of the sediment core. The length of the core 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.

A14. Compare the length of the recovered core with the core penetration depth.

- If the recovered length of the sediment core is more than 80% of the penetration depth, keep the core.
- If insufficient amount of material is recovered, set the intact core tube aside in an upright position for potential future use, and perform the following steps as necessary.
  - An additional attempt will be made at a minimum distance of 2 feet from the previously attempted location.
  - A maximum of three attempts to collect a core will be made for a given location.
  - If all three attempts to collect a core are unsuccessful based on recovery alone (i.e., less than 80% recovery), retain the core with greatest recovery and indicate that the targeted recovery was not achieved (dispose other cores).
  - If three attempts to collect a core are unsuccessful in recovering any sediment, attempt collection with a hand-held ponar dredge (see C1 through C9 below). If no sediment can be collected via this method, document collection attempts and abandon the location.

A15. Remove the core tube from the vibracore 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 water.

A16. After successful core recovery enter additional information into the field database: 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.

A17. Draw an arrow on the core tube or write "top" on the core tube with a paint marker to mark the top of the core. Label the core with paint marker indicating station ID, date, and time.

A18. Store the core vertically in the specially designed core holder while on the vessel and transport to the off-loading area for subsequent transport to the processing area.

A19. Upon delivery of the core 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. At this time, compare the field collection recovery to the processing lab recovery to confirm that voids in the recovered core tube were not present as evidenced by settling in the core tube. Any core with a 1 inch per foot difference will be discarded and re-collected.

A20. Due to the potential for soft/loose surface sediment, the upper sections (to one foot depth) may need to be sectioned using a pipe cutter (or other appropriate device) with the core kept in a vertical position and locked in a core stand. Each of these sections will be carefully transferred into a disposable aluminum pan for further processing.

A21. For lower sample sections or cores with consolidated surface sediment, 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.

A22. Physical descriptions of each core will be documented and changes in stratigraphy noted. Characteristics include the general soil type based on the Unified Soil Classification System, approximate grain size (fine,

medium, and coarse), presence of observable biota, odor, and color. Obtain photographs of the sediments at a maximum of 2 foot increments alongside a measuring tape.

A23. Remove representative samples based on the segmentation scheme outlined in the CQAP.

A24. Fully homogenize samples until a uniform color and consistency is obtained.

A25. Place the homogenized sample in appropriate sample containers and cap.

A26. Label all sample containers.

A27. Place filled sample containers on ice in a cooler or specifically designated refrigerator/freezer per the QAPP Worksheets preservative methods.

A28. Quality assurance/quality control samples will be obtained as specified in the QAPP Worksheets.

B. Sediment Sampling via Manual Coring

Materials
The following materials will be available, as required, during sediment sampling via manual coring:

- Health and safety and personal protective equipment (as required by the Health and Safety Plan [HASP], Contractor Safety Work Plan [CSWP], and associated Job Safety Analyses [JSAs])
- Anchor/concrete blocks
- Boat and motor
- Rope
- 3” aluminum tubing or Lexan® tubing with end caps
- Calibrated probe rod
- Surveyor's rod
- Hacksaw
- Steel core driver
- Piston sampler/check valve push core device
- RTK survey equipment
- Disposable aluminum pans
- Disposable spatulas
- Appropriate sample containers and forms
- Coolers with ice
- Field notebook and computer
- Camera
- Duct tape
- Six-foot rule

Sediment Sampling Procedures
As an alternate sampling method in the event core collection is unsuccessful or not possible via vibracoring, steps A7 through A11 will be replaced with steps B1 through B6 below, for collection of manual sediment cores from a boat.

B1. Once the targeted area is deemed suitable for core collection, select an appropriate length of 3-inch outside diameter aluminum core tube or 3-inch outside diameter Lexan® tube based on the probing information.

B2. Lower the section tube vertically until it just reaches the top of the sediment. Sections of tube may need to be spliced together or the tube may be attached to a check valve core device.

B3. Push the tube with a straight vertical entry into the sediment so as to secure a reliably representative core sample. Measure and record the depth of core tube penetration into the sediments in the field book.

B4. Drive the tube until refusal using a steel core driver and measure the additional distance. This procedure is performed to obtain a "plug" at the bottom of the core and prevent loose sediment from escaping.

B5. Fill tube with river water and place an end cap on the top end of the tube and create a vacuum to prevent the sediment from escaping (note this is not needed if using a check valve core device).

B6. Slowly pull the tube from the sediment, twisting it slightly as it is removed (if necessary) and secure with an end cap before it breaks the water's surface.

C. Sediment Sampling Using a Hand-Held Dredge

Materials
The following materials will be available, as required, during sediment sampling via hand-held dredge:

- Health and safety and personal protective equipment (as required by the Health and Safety Plan [HASP], Contractor Safety Work Plan [CSWP], and associated Job Safety Analyses [JSAs])
- Anchor/concrete blocks
- Boat and motor
- Rope
- Hand-held dredge (standard 6" petite ponar)
- Recovery winch if needed
- Decontamination solvents (hexane or alconox) and distilled water
- Calibrated probe rod
- Surveyor's rod
- RTK survey equipment
- Disposable aluminum pans with aluminum lids
- Disposable spatulas
- Appropriate sample containers and forms
- Coolers with ice
- Field notebook and computer
- Camera
- Duct tape

Sediment Sampling Procedures
As an alternate sampling method in the event core collection is unsuccessful in recovering sediment, steps A7 through A24 may be replaced with steps C1 through C8 below, for collection of grab sediment samples through use of a hand-held ponar dredge.

C1. At each sample location, slowly lower open dredge from the side of the boat making sure that the end of the rope is maintained at all times until just above sediment surface and then drop the open dredge into the sediment.

C2. Once the dredge has been allowed to settle into the bottom sediment, pull hard on the rope to close the sediment inside the dredge.

C3. Retrieve the dredge onto the boat utilizing the recovery winch as needed.

C4. Inspect the sample for acceptability. The following acceptability criteria should be satisfied:

- The sampler is not overfilled with sample such that the sediment surface presses against the top of the sampler or is extruding through the top of the sampler.
- Overlying water is present (indicates minimal leakage).
- The overlying water is not excessively turbid (indicates minimal disturbance or winnowing).

C5. Tilt dredge slightly to drain overlying water and then open the dredge to allow the sediment to empty onto a disposable aluminum pan.

C6. If needed, multiple casts may be made and composited at each location until sufficient sample volume is obtained.

C7. Clean the sampling equipment prior to initial use, between sampling at each location, and at the completion of sampling. Specifically, clean the dredge between sample locations as follows: distilled water rinse; hexane or alconox triple rinse; and distilled water triple rinse; allow to air dry; and wrap in aluminum foil.

C8. Obtain rinse blanks as specified in the QAPP Worksheets by pouring de-ionized water through a cleaned stainless steel dredge and fill the appropriate sample containers.

Standard Operation Procedure #2: Floodplain Soil Sampling

The protocol set forth in this Standard Operating Procedure (SOP) outlines the procedures for floodplain soil sampling using hand tools (i.e., aluminum scoop or coring device). Details regarding the sampling objectives, design, and quality assurance requirements are provided in the Construction Quality Assurance Plan (CQAP) and Quality Assurance Project Plan (QAPP) Worksheets (Appendix C); the procedures outlined here will be performed in compliance with the CQAP and QAPP Worksheets.

Materials:

The following materials will be available, as required, during floodplain sampling:

- Health and safety and personal protective equipment (as required by the Health and Safety Plan [HASP], Contractor Safety Work Plan [CSWP], and associated Job Safety Analyses [JSAs])
- Real-Time Kinematic (RTK) survey equipment
- Decontamination solvents (hexane or alconox) and distilled water
- Disposable aluminum pans
- Field notebook and computer
- Appropriate sample containers and forms
- Stainless steel trowel, scoop, and hand auger
- Macrocore® sampler
- Slide hammer
- Engineer's ruler
- Coolers with ice
- Aluminum or stainless spade, spoon, scoop, or hand auger
- Disposable spatulas
- Camera

Floodplain Soil Sampling Procedures

The following procedures will be employed to collect floodplain surface soil samples (i.e., top 6 inches). Samples will be collected at designated locations.

1. Don health and safety equipment (as required in the HASP, CSWP, and JSAs).

2. Setup RTK survey equipment (includes master station and rover). Set master station on known control point and check coordinates. Set rover on different control point and check coordinates. Setups are considered acceptable if coordinates check to within 0.1 feet horizontally and vertically. If not within accuracy, perform troubleshooting efforts including verifying control point coordinates and numbers, confirm no physical impacts to control points, etc. If accuracy cannot be achieved, utilize different control point(s). The rover station will be used to navigate to the sample location.

3. Locate the target sample location using RTK surveying techniques.

4. Clear the ground surface of brush, root mat, grass, leaves, or other debris if present.

5. Use a spade, spoon, scoop, or hand auger to collect a sample of the required depth (i.e., top 6 inches).

6. Use an engineer's ruler to verify that the sample is collected to the correct depth and record the top and bottom depths from the ground surface.

7. Backfill sample holes to grade with native material or with clean builder's sand or other suitable material.

8. Prepare a clean set of disposable aluminum pans for the soil sample and place the sample from the spade, spoon, scoop, or hand auger into a pan.

9. Record physical descriptions of the sample. Characteristics include the general soil type based on the Unified Soil Classification System (USCS), approximate grain size (fine, medium, and coarse), odor, and color. Obtain photographs of the sample prior to homogenization.

10. Fully homogenize the sample in the disposable aluminum pan until a uniform color and consistency is obtained.

11. Place the homogenized sample in the appropriate sample container and cap.

12. Label all sample containers.

13. Place filled sample containers on ice in a cooler or specifically designated refrigerator/freezer per QAPP Worksheets preservative methods.

14. Clean the sampling equipment prior to initial use, between sampling at each location, and at the completion of sampling. Specifically, clean the dredge between sample locations as follows: distilled water rinse; hexane or alconox triple rinse; and distilled water triple rinse; allow to air dry; and wrap in aluminum foil.

15. Obtain rinse blanks as specified in the QAPP Worksheets by pouring de-ionized water through the cleaned stainless steel equipment and fill the appropriate sample containers.

16. Quality assurance/quality control samples will be obtained as specified in the QAPP Worksheets.

As an alternate sampling method for those collection locations where deeper soil sampling is necessary (i.e., beyond surficial soil) or if collection with a scoop is not possible, steps 3 through 8 will be replaced with A through J for collection using manual coring techniques.

A. At the designated sampling locations, soil samples will be collected manually using a Macrocore® sampler advanced with a slide hammer. The Macrocore® sampler consists of an outer steel barrel with an inner acetate liner that will be replaced for each new sample. The shoe (drive tip) of the Macrocore® will be decontaminated prior to its initial use, between each sampling location, and at the completion of sampling with a distilled water and hexane or alconox triple rinse. The sampler will be advanced to the required sampling depth.

B. If the sample depth is not reached due to refusal or encountered debris, up to 3 attempts will be made with the Macrocore® sampler in the general area. Then a hand auger will be utilized as a last option to meet the sample depths.

C. Once the acetate liner is extracted from the sampler, use an engineer's ruler to verify that the sample recovery depth is sufficient. Recollect the sample if poor sample recovery (less than 80%) is encountered. If insufficient amount of material is recovered, set the intact core aside in an upright position for potential future use, and perform the following steps as necessary.

- An additional attempt will be made at a minimum distance of 2 feet from the previously attempted location.
- A maximum of three attempts to collect a core will be made for a given location.
- If all three attempts to collect a core are unsuccessful based on recovery alone (i.e., less than 80% recovery), retain the core with greatest recovery and indicate that the targeted recovery was not achieved (dispose other cores).

D. Record field collection information.

E. Label the core tube with the appropriate sample nomenclature, date, time of collection, soil penetration, and recovery.

F. Soil cores will be kept upright and transported to the processing area for documentation, segmentation, and sampling.

G. Acetate soil liners will be split open utilizing a safety cutter on a core processing table.

H. An engineer's ruler will be used to verify that the sample recovery depth matches the noted field collection recovery. If any discrepancies due to voids or settlement are encountered, the sample will be recollected.

I. Physical descriptions of each core will be documented and changes in stratigraphy noted. Characteristics include the general soil type based on the Unified Soil Classification System, approximate grain size (fine, medium, and coarse), presence of observable biota, odor, and color. Obtain photographs of the soils at a maximum of 2 foot increments alongside a measuring tape.

J. Remove representative samples based on the segmentation scheme outlined in the CQAP and placed into disposable aluminum pans for homogenization using disposable wooden tongue depressors. In situations where compression of soil is evident (recovery less than 100% but greater than 80%) within the acetate liner, the processor will make a professional judgment as to the interval within the liner as it relates to the in-situ interval. This will be documented.

Standard Operating Procedure #3: Water Monitoring

The protocol set forth in this Standard Operating Procedure (SOP) outlines the field procedures for water monitoring including the water column and water intake monitoring. Details regarding the sampling objectives, design, and quality assurance requirements are provided in the Environmental Monitoring Plan (EMP) and associated Quality Assurance Project Plan (QAPP) Worksheets; the procedures outlined here will be performed in compliance with the EMP and QAPP Worksheets.

A. Water Column Sampling Procedures

Materials
The following materials will be available, as required, during water column sampling:

- Health and safety and personal protective equipment (as required by the Health and Safety Plan [HASP], Contractor Safety Work Plan [CSWP], and associated Job Safety Analyses [JSAs])
- Appropriate water sampler (e.g., Wildco Kemmerer stainless steel bottle sampler [Kemmerer])
- Decontamination solvents (hexane, alconox, or nitric acid) and distilled water
- Boat and motor
- Surveyor's rod
- Depth sounder
- Combination water quality meter and associated calibration kit (YSI 6920)
- Appropriate field forms/notebook and computer
- Laboratory supplied glassware
- Cooler and ice

Procedures
Water column monitoring will be performed mid channel at the locations specified in the EMP. The procedures for collection of water column samples and water quality parameters (e.g., temperature and specific conductivity) are provided below. Note that water quality parameter readings will be obtained using real-time meters (YSI 6920) from the boat; procedures for setting up, maintaining, and calibrating the meter will be established following in accordance with manufacturer recommendations and user manual.

A1. Don health and safety equipment (as required in the HASP, CSWP, and JSAs).

A2. Identify the sampling location in field forms or notebook along with other appropriate information.

A3. Decontaminate the sampling equipment prior to initial use, between sampling at each location, and at the completion of sampling. Specifically, clean the sampler between sample locations as follows:

- For PCB/TSS sampling - distilled water rinse; hexane or alconox triple rinse; and distilled water triple rinse; allow to air dry; and wrap in aluminum foil.
- For VOC sampling - distilled water rinse; alconox triple rinse; and distilled water triple rinse; allow to air dry; and wrap in aluminum foil.
- For metals sampling - distilled water rinse; nitric acid triple rinse; and distilled water triple rinse; allow to air dry; and wrap in aluminum foil.

A4. Maneuver the sampling vessel to the target sample location.

A5. Measure the total depth of the water column using the depth finder or if shallow enough using a surveyor's rod to the nearest 0.1 foot and record in the field book. If water column depths are less than 5 feet, monitoring will be performed at 0.5 times the total water column depth. For water depths greater than 5 feet, monitoring will be performed at 0.2 and 0.8 times the total water column depth.

A6. Calibrate the water quality meter in accordance with the manufacturer's manual prior to use. For stations with water depth greater than 5 feet, collect in-situ measurements using the water quality meter of temperature and specific conductivity at 0.2 and 0.8 times the total water column depth. Record results and assess the presence of river stratification. Stratification will be confirmed when there are differences of 3 degrees Celsius in water temperature or 20 micro Siemens per centimeter in specific conductivity between the two water masses. If stratification is not present, sample volume will be collected from 0.2 and 0.8 times the total water column depth and composited. If stratification is present, monitoring will be altered to only collect samples from 0.2 times the total water column depth. Modifications to this sampling approach may be required to incorporate the sample from 0.8 times the total water column depth if needed as part of an advisory level or corrective action level assessment.

A7. Lower the water sampler (Kemmerer) to the target sample depth and release trigger mechanism. Raise the water sampler to the surface with minimal disturbance.

A8. Remove the covers from the appropriate laboratory supplied containers and slightly tilt the mouth of the container below the sampling device.

A9. Empty the sampler slowly, allowing the sample stream to flow gently down the side of the sample container(s) (with minimal entry turbulence).

A10. Secure all sample jar caps tightly.

A11. Label all sample containers.

A12. Place filled sample containers on ice in a cooler.

A13. Repeat steps A7-A12 for collection at the needed additional water depths.

A14. Repeat steps A2-A13 for sample collection and monitoring at each location.

A15. Quality assurance/quality control samples will be obtained as specified in the EMP.

B. Water Intake Sampling Procedures

Materials
The following materials will be available, as required, during water intake sampling:

- Health and safety and personal protective equipment (as required by the Health and Safety Plan)
- Appropriate turbidity meter (LaMotte)
- Submersible pump/tubing
- Vinyl tubing
- Duct tape
- Appropriate field forms/notebook and computer

- Laboratory supplied glassware
- Cooler and ice

Procedures
Water intake monitoring will be performed at the locations identified in the EMP. The procedures for collecting water intake samples and turbidity readings are provided below.

B1. Don health and safety equipment (as required in the HASP, CSWP, and JSAs).

B2. Identify the sampling location in field forms or notebook along with other appropriate information.

B3. Perform monitoring at designated locations in coordination with treatment plant and/or facility personnel. If location has an established valve/spigot setup, collect water by opening valve; if location does not have an established valve/spigot setup sample, use a submersible pump/tubing to collect water.

B4. Remove the covers from the appropriate laboratory supplied containers and slightly tilt the mouth of the container below the valve/spigot or pump/tubing setup.

B5. Allowing the sample stream to flow gently down the side of the sample container(s) (with minimal entry turbulence).

B6. Secure all sample jar caps tightly.

B7. Label all sample containers.

B8. Place filled sample containers on ice in a cooler.

B9. Calibrate the turbidity meter in accordance with the manufacturer's manual prior to use. Fill the vial provided with the turbidity meter using the same process outlined in steps B4 and B5. Insert the vial into the meter in accordance with product manual instructions to obtain the turbidity reading. Record the turbidity value.

B10. Repeat steps B2-B9 for collection at all water intake locations.

B11. Quality assurance/quality control samples will be obtained as specified in the EMP.

Standard Operating Procedure #4: Air Monitoring

The protocol set forth in this Standard Operating Procedure (SOP) outlines the field procedures for air monitoring. Details regarding the sampling objectives, design, and quality assurance requirements are provided in the Environmental Monitoring Plan (EMP) and associated Quality Assurance Project Plan (QAPP) Worksheets; the procedures outlined here will be performed in compliance with the EMP and QAPP Worksheets.

Materials
The following materials will be available, as required, during air monitoring:

- Health and safety and personal protective equipment (as required by the Health and Safety Plan [HASP], Contractor Safety Work Plan [CSWP], and associated Job Safety Analyses [JSAs])
- Appropriate air monitoring equipment (i.e., particulate/VOC meters and low/high-volume samplers) and associated calibration kits and batteries
- Vinyl tubing
- Equipment/sampling tripod
- Appropriate field forms/notebook and computer
- Laboratory supplied media and glassware

Procedures
Air monitoring will be performed at the locations specified in the EMP. The procedures for collection of air samples for PCB analysis and recording particulate/VOC levels are provided below. Samplers/meters will collect data over a 24-hour timeframe, unless the monitoring period is shortened to match the duration of construction activities in accordance with the EMP. Details on air monitoring equipment setup, calibration, and operation are provided in the manufacturer's manuals, and references to these manuals are made throughout the SOP. The air monitoring equipment includes the high-volume sampler (Tisch TE-1000PUF), low-volume sampler (SKC Leland Legacy Pump), particulate meter (DustTrak), and VOC meter (MiniRAE 3000).

1. Don health and safety equipment (as required in the HASP, CSWP, and JSAs).

2. Field reconnaissance and initial setup:

   2.1. Perform field reconnaissance for the fixed air monitoring stations (i.e., adjacent to the staging area and secure landfill) considering the locations targeted in the EMP, power availability, and site conditions. Once suitable locations are identified, deploy the equipment and setup the particulate/VOC meters and high-volume sampling equipment in accordance with the manufacturer's manual. Perform an initial calibration of the equipment in accordance with the manufacturer's manual.

   2.2. Place each particulate/VOC meter set within a weather enclosure and power on the included modem. Coordinate data reporting to the online telemetry system in coordination with the equipment provider/telemetry technical support. Ensure that early warning and action email alarms are applied and functional for each unit.

   2.3. Evaluate the remedial construction contractor's operations and anticipated initial near shore work area, and perform field reconnaissance for the mobile air monitoring stations considering the locations targeted in the EMP, the location of the nearest receptor, and access agreements. Once a suitable

location is identified, deploy and setup the low-volume sampling equipment in accordance with the manufacturer's manual. Perform an initial calibration of the equipment in accordance with the manufacturer's manual. Charge all batteries in full prior to deployment. Record station coordinates and location description in the field notebook.

3. During-construction monitoring efforts:

   3.1. Identify station in field forms or notebook along with other appropriate information (i.e., document weather and field conditions).

   3.2. Re-calibrate equipment in accordance with the manufacturer's manual as needed.

   3.3. Set particulate/VOC meter to run continuously and initiate the pumps on the continuous air sampling equipment.

   3.4. Return to station throughout the day to confirm equipment operation and/or troubleshoot as needed.

   3.5. Return to station at specified time (i.e., following the 24-hour monitoring period or at the end of construction efforts for the day). Evaluate power and replace the batteries in the meters as needed. Collect air PCB samples (media) and replace sample media for the next sample collection in accordance with the manufacturer's manual. Record the air volume collected by the continuous air samplers as needed and record in the field forms or notebook. Replace low-volume samplers with a fully charged unit.

   3.6. Wrap all PCB sample media vessels in aluminum foil, bubble bags, and resealable plastic bags. Label all sample containers.

   3.7. Place sampling media on ice in a cooler.

   3.8. Repeat steps 3.1-3.7 at all monitoring stations.

   3.9. Quality assurance/quality control samples will be obtained as specified in the EMP.

Standard Operation Procedure #5: Sample Handling, Packaging, and Shipping

The protocol set forth in this Standard Operating Procedure (SOP) outlines the procedures to be used for sample handling, packaging, and shipping. Sampling details and quality assurance requirements are provided in the Environmental Monitoring Plan (EMP) and associated Quality Assurance Project Plan (QAPP) Worksheets; the procedures outlined here will be performed in compliance with the EMP and QAPP Worksheets.

Handling

The required procedures for sample handling are provided below.

1. Completely fill in or verify the automated label generator has included the following information on the sample label:

    - Sample type (e.g., sediment)
    - Sample identification
    - Required analysis
    - Sample date and time
    - Preservative added (if applicable)

2. Cover the label with clear packing tape to secure it onto the container.

3. Check and secure the caps on the sample containers to ensure tight seal.

4. Initiate chain-of-custody by sampling personnel responsible for sample custody (after sampling or prior to sample packing). Note: If the designated sampling person relinquishes the samples to personnel, the chain-of-custody will be completed prior to transfer. The appropriate personnel will sign and date the chain-of-custody form to document the sample custody transfer.

Packing

The required procedures for sample packing are provided below. These procedures are only necessary for samples being sent from the site to a laboratory via express courier or commercial carrier using a cooler(s).

1. Secure the outside and inside of the drain plug at the bottom of the cooler (if applicable) and place cushioning material at the bottom of the cooler.

2. Place each container or package in individual polyethylene bags (Ziploc®-type), seal, and package upright in the cooler.

3. If preservation on ice is required per the PDI QAPP, package ice Ziploc®-type plastic bags (double bagged) and place in the cooler, and fill the remaining space with cushioning material as necessary.

4. Place the completed chain-of-custody forms in a large Ziploc®-type bag and duct-taped to the inside of the cooler lid.

5. The cooler will be closed and fastened with duct tape around the seam of the lid to prevent water leakage and with strapping or duct tape around the entire cooler to prevent it from opening during transport.

6. A completed custody seal will be placed across the seam of the cooler lid. A completed address label will be placed on top of the cooler. Both will be taped-over using clear packing tape.

Shipping

The required procedures for sample shipping are provided below.

1. All samples will be hand delivered or delivered by an express carrier within the requested timeframe (typically 2-4 hours for the courier and within 24 hours by a commercial carrier).

2. The following chain-of-custody procedures will apply to sample shipping:

    - Relinquish the sample containers to the laboratory via express carrier. The signed and dated forms should be included in the cooler. The express carrier is not required to sign the chain-of-custody forms. The sampler should retain the express carrier receipt or bill of lading.
    - When the samples are received by the laboratory, the laboratory personnel shall complete the chain-of-custody forms.

STANDARD OPERATING PROCEDURE SOP-006 – GRANULAR ACTIVATED CARBON SAMPLE COLLECTION AND TESTING

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June 2019

STANDARD OPERATING PROCEDURE ACKNOWLEDGEMENT FORM

Project No. E91444-01.01 Project Name: Grasse River Sediment Remediation Project

My signature below certifies that I have read, understand, and will follow the procedures specified in this Standard Operating Procedure.

Date	Name (print)	Signature	Company

SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) is applicable to the collection of samples from the chemical isolation layer sand amended with granular activated carbon (GAC), Type A Backfill amended with GAC, and Type B Backfill amended with GAC and the testing of these samples for dry weight GAC content for work conducted on the Grasse River Sediment Remediation Project for the 2018 and 2019 Staged Capping Test, 2019 backfill placement, and full-scale cap placement (2020 and 2021). During various phases of the project, samples of the chemical isolation layer (sand with GAC), Type A Backfill amended with GAC, and Type B Backfill amended with GAC (referenced herein as "placement material amended with GAC") will be tested prior to placement (ex situ) and after placement (in situ). Pre-placement samples will be collected following a modified version of American Society for Testing and Materials (ASTM) D75-14 (Standard Practice of Sampling Aggregates). Post-placement samples will be collected using catch pans or similar equipment. Procedures for both pre- and post-placement sample collection are described herein.

In conjunction with the Staged Capping Test, analysis of dry weight GAC content will be conducted using two different methods - a Thermal Drying Method and a Heavy Liquid Separation (HLS) Method. The Staged Capping Test will act as a trial run to evaluate which approach to testing methodologies will be used for the remainder of the project.

In conjunction with backfill placement, analysis of dry weight GAC content will be conducted using the HLS Method.

Procedures in this SOP will be followed to verify the GAC content in placement material amended with GAC. If proposed, substantive deviations from the procedures detailed in this SOP will be discussed and agreed upon with the Construction Manager and the Engineer prior to implementation.

HEALTH AND SAFETY WARNINGS

Each person executing these procedures must read, be familiar with, and comply with the requirements of this SOP, their company's respective Health and Safety Plan (HASP), and the Pre-Design Investigation (PDI) Quality Assurance Project Plan (QAPP; Alcoa, October

2013). Field personnel must be under the direct supervision of qualified professionals who are experienced in performing the tasks required for sample collection and testing. Note that worksheets specific to GAC sample collection and testing were not prepared in the PDI QAPP or associated addenda; the worksheets have since been developed and are presented in Attachment C of the Construction Quality Assurance Plan (Appendix C to the Final Design Report).

EQUIPMENT AND SUPPLIES

The following is a list of equipment that may be necessary to carry out the procedures in this SOP. Additional equipment may be required, depending on conditions in the field.

- Sampling vessel equipped with necessary differential global positioning system (DGPS) navigation and communication equipment
- Approved documents, including this SOP, the PDI QAPP, and the HASP
- Catch pan(s) for sample collection (along with necessary retrieval lines and buoys)
- Polycarbonate (e.g., Lexan®) core tubes and caps
- Tubing cutters
- 2- or 5-gallon buckets with lids
- Aluminum mixing tray
- Sample jars, 4 ounces or larger
- Permanent marker
- Shovel
- No. 10 sieve
- No. 50 sieve
- Sampling bowls
- High-temperature oven
- Duct tape
- Chain-of-custody forms
- Field notebook
- Appropriate personal protective equipment and clothing as defined in the HASP

SAMPLING PROCEDURE FOR PRE-PLACEMENT SAMPLE COLLECTION

Samples of the chemical isolation layer sand with GAC will be collected at the frequency described in Specification Section 31 23 23 – Capping and Backfilling, or as otherwise agreed to with the Construction Manager and the United States Environmental Protection Agency. Sampling of Type A or Type B Backfill will be conducted once a day during GAC amended backfill placement. Sampling procedures will follow a modified version of ASTM D75-14 and the instructions herein.

1. Samples will be collected in clean 2- or 5-gallon buckets using the approach described in ASTM D75-14. A minimum of 8 kilograms (kg; approximately 18 pounds) of material is to be collected for each sample tested. Samples of pre-placement (ex situ) GAC amended material will be collected from a conveyor belt used to blend and transport the amended material. For the Staged Capping Test sand-only samples will be collected from a sand stockpile coincident with the collection of ex situ amended material samples. The following procedure summarizes the sampling process based on ASTM D75-14:
    a. Obtain at least three approximately equal increments, selected at random, from the conveyor belt or stockpile.
    b. Combine the collected amended material to form a field sample with a mass of 16 kg (approximately 35 pounds) or more. Note, a minimum total of 16 kg is needed to run triplicates for the Thermal Burn Method. A total of 12 ounces (split into three 4-ounce jars) is required for the HLS Method, if needed.[1]
    c. Homogenize field sample with shovel (or similar tool)
    d. Fill the sample jar (minimum 4-ounce size) with the homogenized sample. The rest of the sample shall remain in the bucket with the lid on.
2. Label the bucket and the sample jar as follows: GREX-MATRIX-YYYYMMDD-SAMPLE#.
    a. Where:
        i. "GREX" stands for Grasse River ex situ
        ii. "MATRIX" is the matrix code for either:

[1] Samples collected during the Staged Capping Test and initial rounds of backfill placement will be tested in triplicate. Depending on those results, testing volume may be adjusted for full-scale capping operations and backfill placement (i.e., testing in triplicate may not be required).

1. "SA" = Sand
2. "SAG" = Sand with GAC
3. "TAG" = Type A Backfill with GAC
4. "TBG" = Type B Backfill with GAC

iii. "YYYYMMDD" refers to the year, month, and day of sample collection

iv. The sample number increases by one from the start of each construction season

3. Log sample collection notes in field notes and create a chain-of-custody form for each laboratory needed prior to shipment or delivery.
   a. Sample jars to be submitted for analysis via the HLS Method will be sent to Anchor QEA's Portland, Oregon, office. Chain-of-custody forms and sample jars will be sent via FedEx.
   b. Sample buckets to be submitted for analysis via the Thermal Drying Method will be delivered to Atlantic Testing Laboratories' (ATL's) East Syracuse, New York, office, or to an on-site laboratory if available. Chain-of-custody forms and buckets will be delivered via FedEx or by courier.

Refer to ASTM D75-14 for alternative methodologies for securing samples from various source streams.

SAMPLING PROCEDURE FOR POST-PLACEMENT SAMPLE COLLECTION

Post-placement samples of the chemical isolation layer (i.e., sand with GAC) will be collected, if required,[2] as described in Specification Section 31 23 23 – Capping and Backfilling by catch pans as the primary sampling method. During the Staged Capping Test, each homogenized sample will be created by compositing four individual catch pan samples. Additional collection methods may be used but shall be agreed upon with the Construction Manager and the Engineer as described in the previous section.

[2] Pre- and post-placement results of Staged Capping Test samples will be evaluated to determine the appropriate sampling methodology to be used during full-scale capping.

Catch pan collection from the chemical isolation layer will be performed using the following procedures:

1. Catch pans will be placed in the river at approved sample locations prior to the placement of cap chemical isolation layer material. Catch pans will be affixed to a retrieval line for future retrieval. Record catch pan location coordinates.
2. After material placement, a vessel will be navigated to the catch pan locations. Record post-placement catch pan locations to confirm catch pans have not shifted.
3. Retrieve each catch pan from the bottom of the river via the affixed retrieval line.
4. Measure the thickness of material collected within the catch pan.
5. Secure the catch pan and transport it to the processing area.
6. Homogenize the contents of the catch pan with a shovel (or similar tool).
7. For individual catch pan samples, manage the sample as described in Step 8. For composite catch pan samples, comprising multiple catch pan samples combined into a single sample for laboratory analysis, manage the sample(s) as described in Step 9.
8. For individual samples, prepare the individual sample as follows:
    a. Place the homogenized catch pan sample into a clean 2- or 5-gallon bucket. The container shall be labeled with catch pan ID (see Step 10).
    b. Fill a sample jar (minimum 4-ounce size) from the homogenized sample. The rest of the sample shall remain in the bucket with the lid on.
    c. Label the bucket and the sample jar as follows: GR-Location#-MATRIX-YYYYMMDD-SAMPLE#, where:
        i. “GR” stands for Grasse River
        ii. “Location#” is the test cell ID (i.e., catch pan location TC-15.3Sa would be TC15.3Sa) or associated Cap Certification Unit. The letter at the end of the “Location#” designates each discrete catch pan collected from a test cell, with “a” being the first catch pan retrieved from a test cell, “b” the second, and so on.
        iii. “MATRIX” is the matrix code for either:
            1. “SAG” = Sand with GAC
        iv. “YYYYMMDD” refers to the year, month, and day of sample collection
        v. The sample number increases by one from the start of each construction season.
9. For composite samples, prepare the composite sample as follows:

a. Collect an aliquot sample from each homogenized catch pan. Place the aliquot sample in a clean aluminum mixing tray.
b. Homogenize the multiple catch pan samples with a shovel (or similar tool).
c. Place the contents of the homogenized composite sample into a clean 2- or 5-gallon bucket. The container shall be labeled with the composite sample ID (refer to step 9e). The bucket shall contain at least 16 kg (approximately 35 pounds) of sample to allow triplicates to be run for the Thermal Burn Method. A total of 12 ounces (split into three 4-ounce jars) is required for the HLS Method, if needed.
d. Fill sample jar (minimum of 4 ounces) with homogenized composite sample. The rest of the sample shall remain in the bucket with the lid on.
e. Label the bucket and the sample jar as follows: GR-Location#-[Comp]-MATRIX-YYYYMMDD-SAMPLE#, where:
    i. "GR" stands for Grasse River
    ii. "Location#" is the test cell ID (i.e., catch pan location TC-15.3S would be TC15.3S) or associated Cap Certification Unit.
    iii. "Comp" stands for "composite"
    iv. "MATRIX" is the matrix code for either:
        1. "SAG" = Sand with GAC
    v. "YYYYMMDD" refers to the year, month, and day of sample collection
    vi. The sample number increases by one from the start of each construction season

10. Log sample collection notes in field notes and create a chain-of-custody form for each laboratory needed prior to shipment or delivery.
    a. Sample jars to be submitted for analysis via the HLS Method will be sent to Anchor QEA's Portland, Oregon, office. Chain-of-custody forms and sample jars will be sent via FedEx.
    b. Sample buckets to be submitted for analysis via the Thermal Drying Method will be delivered to ATL's East Syracuse, New York, office, or to an on-site laboratory if available. Chain-of-custody forms and buckets will be delivered via FedEx or by courier.

Personnel will record field conditions and notes in a log book or database system. Details from the log book will be provided to the Construction Manager. The logs will include the following information:

- Sample location identification
- Coordinates for the sample locations as determined by DGPS
- Date of sample collection
- Names of field personnel collecting and handling the samples
- Observations made during sample collection, including weather conditions, complications, and other details associated with the sampling effort
- Thickness of material in catch pan
- Note whether GAC was observed in the sample
- Date of sample shipment to the laboratory

TESTING FOR DRY WEIGHT GAC CONTENT

As described in the previous section, testing for dry weight GAC content will be performed using two methods, a Thermal Drying Method and an HLS Method. The procedure for the HLS tests is presented in Attachment 1.

Thermal testing will use the approach described by ASTM D2974-14 Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils by Method A for moisture content (or ASTM D2166-19 Standard Test Methods for Laboratory Determination of Water [Moisture] Content of Soil and Rock by Mass).

1. Chemical isolation layer sand with GAC samples will be split into triplicate, weighed, then placed in an oven and heated to 110°C [ASTM D2974 Method A] to remove the water from the sample. Samples will remain at 110°C for at least 2 hours or until moisture removal can be visually confirmed. Pre-drying sample size will be determined such that the resulting sample dry weight is approximately 2 kg.
2. Once the water has been removed, the sample will be weighed then sieved through a U.S. Standard No. 10 sieve, then through a U.S. Standard No. 50 sieve. GAC is not anticipated to pass through the No. 50 sieve given the material's particle size specifications.

3. The sample remaining on the No. 50 sieve will be weighed and placed in an oven and heated to 440°C [ASTM D2974 Method C] to burn off the naturally occurring organics typically found in sand aggregate. Samples will remain at 440°C for a minimum of 3 hours.
4. When complete, the sample will be reweighed to determine the mass of naturally occurring organics that will have burned off during the heating process. The mass of naturally occurring organics will be calculated as the difference between the sample weight from step 2 and the weight from this step and will include a correction factor for natural organics that will remain after this step, based on the testing of control (i.e., sand-only) samples. A correction factor will also be applied for GAC burned off during step 4 based on the results of spiked control sample testing.
5. The sample will be placed in an oven and heated to 620°C to burn off the GAC. Samples will remain at 620°C for a minimum of three hours or until GAC is no longer visible in the sample.
6. The sample will then be reweighed. The mass of GAC in the sample will be calculated as the difference between the weight from step 4 and this step (step 6). Determination of the GAC content on a percent by weight basis will be based on mass relationships using dry weight results (measured at the end of step 1) from the sample masses prior to and after heating while factoring in inherent background material, GAC burned off during the 440°C burn, tare (pan) weight differences at varying temperatures, and ash correlation, which were developed during control sample testing.

The preceding process is subject to revision upon further laboratory testing.

QUALITY ASSURANCE/QUALITY CONTROL

Entries in the field forms will be double-checked by the field team staff to verify that the information is correct. It is the responsibility of the Field Lead to periodically check to ensure that the procedures are in conformance with those stated in this SOP.

REFERENCES

Each company's Health and Safety Plan (HASP).

Alcoa, October 2013. *Pre-Design Investigation Quality Assurance Project Plan*.

Anchor QEA Engineering, PLLC, October 2018. *Grasse River Staged Capping Test – Health and Safety Plan*.

ASTM (ASTM International), 2014. *ASTM D75-14 – Standard Practice for Sampling Aggregates*.

ASTM, 2014. *ASTM D2974 – 14 – Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils.*

ASTM, 2019. *ASTM D2166-19 - Standard Test Methods for Laboratory Determination of Water [Moisture] Content of Soil and Rock by Mass*.

ATTACHMENTS

Attachment 1 – Density Separation: GAC Confirmation

ATTACHMENT 1
DENSITY SEPARATION: GAC CONFIRMATION

DENSITY SEPARATION: GAC CONFIRMATION

Scope and Application

This Standard Operating Procedure (SOP) is applicable to confirmation of weight percent granular activated carbon (GAC) in a reactive cap mixture. GAC is recovered by density separation from the matrix followed by determination of mass on a dry weight basis. Procedures outlined in this SOP are expected to be followed.

Health and Safety

All laboratory work will be performed in accordance with the EGL Chemical Hygiene Plan (CHP) by staff approved to work in the laboratory. Approval to work in the EGL requires orientation to laboratory safety procedures and potential hazards under the guidance of the Laboratory Manager, as specified in the CHP.

Equipment and Supplies

The following is a list of equipment that may be necessary to carry out the procedures contained in this SOP. Additional equipment may be required.

- Heavy liquid solution of appropriate density
- Drying oven 110 degrees Celsius (°C)Standard sieve set
- Precision scale appropriate for mass range
- Stainless steel spoon
- Disposable 3 mL pipettes
- Narrow flasks, jars, or falcon tubes
- Aluminum weigh dishes
- 500-1000 mL beakers
- Funnel
- Paper filters (such as coffee filters)

Procedure

Method Preparation

1. Perform microscopy on pre-mix sand matrix and GAC
2. If grain size has not been determined, perform grain size analysis in accordance with the EGL Grain Size SOP
3. Generate triplicate test samples by combining a known mass of GAC into the sand matrix at a percent mass similar to the expected cap mixture
4. Perform the separation protocol on the test samples
5. Dilute the heavy liquid to the appropriate density with distilled water
    a. Confirm that the unmixed GAC floats on the prepared solution
    b. Confirm that the sand matrix sinks in the prepared solution (and note organic matter contribution from the matrix, if any)
    c. Photo-document the confirmation tests

GAC Recovery Procedure

1. Collect a representative sample (test mixture, recovered core interval) and homogenize
2. Label aluminum weigh boats and record the mass
3. Collect test samples (fill an aluminum weigh dish) in duplicate (or triplicate)
4. Oven-dry the sample for approximately 4 hours at 110 °C
    a. After 2-3 hours of drying, record the mass of 2-3 samples
    b. Allow to dry for an additional 30-60 minutes
    c. Record the mass
    d. Samples are dry when there is no appreciable mass change (e.g., <1% mass change) between measurements
5. Weigh and record the total sample mass
6. Sieve to separate grains larger or smaller than the GAC grain-size range (e.g., #10 and #200 sieve, retaining the fraction on the #200 sieve)
7. If extensive fines are present, wet-sieve the material (selected sieve should retain 99 percent or greater GAC based on previously determined grain sizes)

8. Mix the sample with the heavy liquid solution in a falcon tube or narrow diameter container. (The container should allow approximately 1 inch of clear solution between the GAC and the heavier material)
9. Stir the material thoroughly to release any grains trapped under heavier material
10. Two distinct layers will form, with the GAC grains floating on the top of the solution. If there is not a clear separation, add fresh density solution until there is a clear separation between the two layers.
11. Scoop the GAC layer into a large weigh boat. A small stainless steel spoon works well for this step. Use a disposable pipette to recover any remaining grains
12. Add a fresh aliquot of heavy liquid solution to the sample and stir to confirm all GAC has been recovered
13. Wash the GAC into a large beaker and rinse 5x with an excess of tapwater
    a. Add tapwater until the beaker is 2/3 full
    b. Swirl
    c. Pour the water off into a second beaker (confirm that no sample particles transferred to the second beaker)
14. Rinse 3x with distilled water
15. Transfer the recovered GAC into the original (rinsed) weigh boat and return to the drying oven
16. Oven-dry the sample for approximately 4 hours at 110 °C
    a. After 2-3 hours of drying, record the mass of 2-3 samples
    b. Allow to dry for an additional 30-60 minutes
    c. Record the mass
    d. Samples are dry when there is no appreciable mass change (e.g., <1% mass change) between measurements
17. Record the final GAC mass
    a. Be vigilant for loss of GAC, especially during the extensive rinsing process. Any sample with noted loss of GAC should be excluded from the results.
18. Perform microscopy to confirm recovered GAC
19. Recover the heavy liquid solution by filtration (a coffee filter works well)

Percent Weight GAC

The percentage GAC on a dry weight basis is determined as:

$$100 * \frac{GAC\ (g)}{M_i}$$

GAC= Dry mass GAC recovered by density separation (g)
M_i = Total initial sample mass on a dry weight basis (g)

Quality Assurance/Quality Control

Duplicate or replicate samples should be analyzed at project appropriate rate, with at least one duplicate or replicate sample per event. Where possible this protocol should be performed in duplicate for all samples due to the danger of sample loss during the many processing steps.

The reproducibility is determined for duplicate samples as:

$$RPD = \frac{(X_1 - X_2) \times 100}{(X_1 + X_2) \div 2}$$

RPD = relative percent difference
X_1 = larger result value
X_2 = smaller result value

The precision of the analysis is determined as the relative standard deviation of the test samples:

$$RSD = \frac{s}{\bar{X}} \times 100$$

RSD = relative standard deviation
s = standard deviation
$\bar{X}$= mean of replicates

Data Sheet

Sample	Replicate	Weigh Boat (g)	Total Dry Mass + Boat (g)	GAC Dry Mass + Boat (g)

Table notes
Record sample mass to a precision of 0.001 g for samples less than 100 g

QAPP Worksheet #1 and #2: Title and Approval Page

Site Name/Project Name:	Grasse River Superfund Site
Site Location:	Grasse River Study Area, Massena, New York
Superfund Site Identification Number:	NYD980506232
Document Title:	Pre-Design Investigation Quality Assurance Project Plan (PDI QAPP) – Construction Quality Assurance Plan – Grasse River Study Area

Lead Organization:	Arconic Inc. (Arconic)	
Lead Organization Project Manager:	Lawrence J. McShea	
	Signature	Date
Quality Manager:	Delaney Peterson	
	Signature	Date
Investigation Manager:	Michael S. Schultz	
	Signature	Date
Document Preparer:		
	Michael S. Schultz	
	Signature	Date

Federal Regulatory Agency:	United States Environmental Protection Agency (USEPA)
Federal Regulatory Agency Project Manager:	Young Chang

State Regulatory Agency:	New York State Department of Environmental Conservation (NYSDEC)
State Regulatory Agency Project Manager:	David Tromp

Tribal Environmental Regulatory Agency:	Saint Regis Mohawk Tribe (SRMT) Environment Division
Tribal Regulatory Agency Project Manager:	Jay Wilkins

Applicable Plans and Reports:	• Record of Decision – Grasse River Superfund Site (USEPA, April 2013) • Remedial Design Plan (Alcoa, July 2013) • Pre-Design Investigation Quality Assurance Project Plan (PDI QAPP; Alcoa, October 2013), PDI QAPP Addendum No. 1 (Alcoa, December 2014), PDI QAPP Addendum No. 2 (Alcoa, September 2015), PDI QAPP Addendum No. 3 (Alcoa, December 2015), PDI QAPP Addendum No. 3A (Alcoa, February 2016), PDI QAPP Addendum No. 5 (Alcoa, August 2016), and PDI QAPP Addendum No. 11 (Arconic, January 2019) • Draft Pre-Design Investigation Data Summary Report (PDI DSR; Alcoa, March 2015) • Draft Preliminary Design Report (Alcoa, June 2015) • Draft Intermediate Design Report (Alcoa, September 2016) • Draft PDI DSR Addendum No. 1 (Arconic, March 2017) • Draft PDI DSR Addendum No. 2 (Arconic, January 2019)

QAPP Worksheet #9: Project Planning Session Summary

QAPP Worksheet #9C1: March 5, 2019 Meeting

Project Name: Grasse River	**Site Name**: Grasse River Superfund Site
Projected Date(s) of Field Efforts: 2019 through 2022	**Site Location**: Massena, New York
Lead Organization Project Manager: Lawrence McShea	

Date of Session: March 5, 2019

Scoping Session Purpose: Review of construction monitoring and verification sampling

Name	Title	Organization	Telephone Number	Email Address	Project Role
Young Chang	Remedial Project Manager	USEPA	212-637-4253	chang.young@epa.gov	USEPA Remedial Project Manager
David Tromp	Remedial Project Manager	NYSDEC	518-402-9786	datromp@gw.dec.state.ny.us	NYS Remediation Project Manager
Kevin Farrar	Case Manager	NYSDEC	518-402-9778	kxfarrar@gw.dec.state.ny.us	NYS Remediation Manager
Scarlett McLaughlin	Public Health Specialist	NYSDOH	518-402-7860	sem10@health.state.ny.us	NYS Public Health Consultant
Jay Wilkins	Arconic Oversight Specialist	SRMT	518-358-5937	jay.wilkins@srmt-nsn.gov	SRMT Representative
John Ciampa	Director of Geophysical Services	Spectra Environmental Services	518-782-0882	jciampa@spectraenv.com	SRMT Technical Consultant
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Lawrence McShea	Program Manager	Arconic	724-337-5458	larry.mcshea@arconic.com	Arconic Project Manager
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Chuck Guest	Senior Managing Engineer	Anchor QEA	518-886-	cguest@anchorqea.com	Arconic Technical Team
Heather VanDewalker	Associate Vice President	ARCADIS	315-671-9382	heather.vandewalker@arcadis-us.com	Alcoa Technical Team
Sarah Hill	Principal Engineer	ARCADIS	315-671-9165	sarah.hill@arcadis-us.com	Alcoa Technical Team

Notes/Comments: Review and open discussion regarding proposed 2019 Snug Harbor geotechnical investigations.

Consensus Decisions Made: Agreement on rationale for additional in-river geotechnical investigations to be conducted near Snug Harbor in 2019.

QAPP Worksheet #11: Project/Data Quality Objectives

QAPP Worksheet #11C1: Task C1 – Post-Dredge/Excavation Sediment and Soil Sampling

TASK C1: POST-DREDGE/EXCAVATION SEDIMENT AND SOIL SAMPLING

Problem Statements and Objectives

The United States Environmental Protection Agency (USEPA) issued a Record of Decision (ROD) that identified the remedy USEPA selected to reduce ecological and human health risks associated with polychlorinated biphenyls (PCBs) in sediments of the lower Grasse River. The remedy includes a combination of sediment and floodplain soil removal and backfilling in the shallow near shore and adjacent areas of the river, armored capping sediments in the ice scour prone portion of the river, and capping in the lower part of the river. Following sediment and soil removal, post-dredge/excavation sampling will be conducted to verify that removal has addressed PCB contamination in the designated areas as outlined in the Construction Quality Assurance Plan (CQAP; Arconic, April 2019).

Information Inputs

All samples will be submitted for analysis of total PCBs (Aroclors) and total solids.

Task Boundaries

Once the cutline criteria has been accepted in the river, sediment cores will be collected at pre-defined sampling locations within each Dredge Management Unit (DMU). The required nominal sampling density will be eight cores per acre of DMU, with locations offset from existing design cores. A greater density of sampling will be employed, where necessary, so that each discrete dredge area within a DMU has at least one sample. Therefore, in cases where a DMU makes up multiple individual design areas, post-dredge sampling will include at least one sample in each area. Cores will be advanced to refusal and segmented in 6-inch intervals. The top three segments (depending on core recovery) will be analyzed for PCBs, and any remaining sections will be archived. Sampling will target recovery down to refusal; however, a minimum recovery of 2 inches will be required.

Per the April 2013 ROD, the near shore areas are defined as the submerged area between the upland and the location where the gentle bathymetric slope along the shoreline meets the steep slope of the main channel side walls (generally these areas have 5 feet or less of water during normal summer flow and extend on average approximately 25 feet from shore).

Similarly, once the floodplain soil area grades have been accepted, samples will be collected at pre-defined sampling locations within each area at a frequency of one sample per approximately 900 square feet, with locations offset from existing design locations. A minimum of one sample will be collected from each area. Samples will be collected from the 6-inch interval immediately below the excavation bottom. Additional 6-inch samples may be collected pending the results of the initial surface samples.

Analytic Approach and Acceptance Criteria

PCB results will be evaluated as outlined in the CQAP. Worksheets #20 and 23 provide the field quality control summary and analytical Standard Operating Procedures (SOPs), respectively. Data will need to be collected and analyzed in conformance with USEPA Region 2 QA guidance and manuals (https://www.epa.gov/quality/region-2-quality-assurance-guidance-and-standard-operating-procedures).

Plan for Obtaining Data

Sediment sampling activities will be conducted consistent with the field sampling SOPs provided in the CQAP. Target locations may be adjusted in the field based on conditions encountered and professional judgment of the field crew conducting the efforts. Analysis design requirements are provided on Worksheets #19, 20, 24, 25, and 30.

QAPP Worksheet #11: Project/Data Quality Objectives

QAPP Worksheet #11C2: Task C2 – Water Quality Monitoring

TASK C2: WATER QUALITY MONITORING

Problem Statements and Objectives

The United States Environmental Protection Agency (USEPA) issued a Record of Decision (ROD) that identified the remedy USEPA selected to reduce ecological and human health risks associated with polychlorinated biphenyls (PCBs) in sediments of the lower Grasse River. The remedy includes a combination of sediment and floodplain soil removal and backfilling in the shallow near shore and adjacent areas of the river, armored capping sediments in the ice scour prone portion of the river, and capping in the lower part of the river. During in-river and adjacent floodplain debris removal, dredging, excavation, backfilling, and capping, water column and water intake monitoring will be performed, as outlined in the Construction Quality Assurance Plan (CQAP; Arconic, April 2019), to assess the potential impacts of remedial activities.

Information Inputs

All water column samples (upstream fixed, near-field, downstream fixed, and far-field stations) will be submitted for analysis of total suspended solids (TSS), water column samples downstream of operations (near-field, downstream fixed, and far-field stations) will be submitted for total PCBs (Aroclors), and all water intake samples will be submitted for total PCBs (Aroclors). In addition, the field crew will record total water column and sample depths at each Grasse River location, as well as water quality information (temperature and conductivity) at stations with water depths greater than 5 feet to check for the presence of stratification. Differences of approximately 3 to 5 degrees Celsius (°C) in water temperature and approximately 20 micro Siemens per centimeter (µS/cm) in specific conductivity between the two water masses (i.e., 0.2 and 0.8 times the total water column depths) will be used to identify the existence of stratification. Real-time measurements of turbidity levels will also be recorded at the time of water intake sampling. Monthly sampling for PCB congener analysis will also be performed at the Saint Regis Mohawk Tribe water intake.

In addition, grab samples will be collected once a week for the first month of near shore dredging, debris removal, and backfilling from the near-field station and submitted for analysis of lead, mercury, and select polycyclic aromatic hydrocarbons (PAHs).

Task Boundaries

Locations for water column monitoring include a fixed location upstream (upstream of remedial action areas), a near-field station (mobile station approximately 1,000 feet downstream of the work areas), a fixed location established at the historic water column sampling location WC013 (located just upstream of the Grasse River mouth at T71), and a far-field station in the St. Lawrence River. At each station, monitoring will be performed at the midpoint of the river near the water surface and the mudline (i.e., 0.2 and 0.8 times the total water column depth) to account for stratification. Grab samples will be composited if stratification is not present. If water depths are shallow (e.g., approximately 5 feet or less), monitoring will be performed mid-depth (0.5 times the total water column depth).

The Arconic East Plant and St. Regis Mohawk Tribe water intakes will also be sampled at the ports within each facility.

Analytic Approach and Acceptance Criteria

Monitoring will be performed during remedial activities to assess the potential impacts of remediation on water quality, as outlined in the CQAP. Worksheets #20 and 23 provide the field quality control summary and analytical SOPs, respectively. Data will need to be collected and analyzed in conformance with USEPA Region 2 quality assurance guidance and manuals (https://www.epa.gov/quality/region-2-quality-assurance-guidance-and-standard-operating-procedures).

Plan for Obtaining Data

Sampling activities will be conducted consistent with the field sampling SOPs provided in the CQAP. Target locations may be adjusted in the field based on conditions encountered and professional judgment of the field crew conducting the efforts. Analysis design requirements are provided on Worksheets #19, 20, 24, 25, and 30.

QAPP Worksheet #12: Measurement Performance Criteria

Matrix	Air
Analytical Group[1]	PCB Aroclors (TO4 and TO10)
Concentration Level	Low

DQIs	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria
Accuracy/Bias/ Sensitivity-Contamination	MB/Instrument Blank	No target compound >QL[2]
Accuracy/Bias	LCS	65%-125% R
Accuracy/Bias/Precision	LCSD	65%-125% R; RPD ≤ 30%
Accuracy/Bias	Surrogates	60%-120% R
Accuracy/Precision	Second column confirmation	40% RPD
Precision	Field Duplicate	RPD ≤50% if both results are >5x QL. Difference ≤QL if results are ≤5x QL.
Completeness	Data Completeness Check	≥90%

Notes:
1. Refer to QAPP Worksheet #15 for a complete list of analytes for each analytical group.
2. If MB exceedances are widespread or reoccurring, analyses must stop and the source of contamination must be eliminated or reduced.
3. Equipment blank contamination will be evaluated on an individual basis.

Acronyms:
DQI: data quality indicator
MB: method blank
QC: quality control
QL: quantitation limit
LCS: laboratory control sample
LCSD: laboratory control sample duplicate
% R: percent recovery
PCB: polychlorinated biphenyl
RPD: relative percent difference

QAPP Worksheet #12: Measurement Performance Criteria

Matrix	Sediment/Soil
Analytical Group[1]	GAC Content (Thermal Burn and Heavy Liquid Separation)
Concentration Level	N/A

DQIs	QC Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria
Completeness	Data Completeness Check	≥90%
Accuracy/Precision	Triplicates	TBD

Notes:
1. Refer to QAPP Worksheet #15 for a complete list of analytes for each analytical group.

Acronyms:
DQI: data quality indicator
GAC: granular activated carbon
N/A: not applicable
QC: quality control
TBD: to be determined after the completion of the Staged Capping Test

QAPP Worksheet #14/16: Project Tasks & Schedule

Activity	Responsible Party	Planned Start Date	Planned Completion Date	Deliverable(s)	Deliverable Due Date
Post-Dredge/Excavation Sediment and Soil Sampling	Arcadis	April 2019	November 2019	Data to be summarized in the 2019 Construction Completion Report	Upon completion of field activities and data validation (deliverable estimated for Q1 2020)
Water Quality Monitoring	Arcadis	April 2019	November 2019	Data to be summarized in the 2019 Construction Completion Report	Upon completion of field activities and data validation (deliverable estimated for Q1 2020)

Acronyms:
Q1 – First Quarter

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

QAPP Worksheet #15E: Air

Analyte	CAS Number	PAL	PAL Reference	Project Quantitation Goal	Achievable Laboratory QLs[a]	Achievable Laboratory DLs[a]
PCBs as Aroclors (µg/m^3)						
Aroclor-1016	12674-11-2	N/A	N/A	0.001	0.00031	0.0000085
Aroclor-1221	11104-28-2			0.001	0.00031	0.0001
Aroclor-1232	11141-16-5			0.001	0.00031	0.000085
Aroclor-1242	53469-21-9			0.001	0.00031	0.00003
Aroclor-1248	12672-29-6			0.001	0.00031	0.000019
Aroclor-1254	11097-69-1			0.001	0.00031	0.000044
Aroclor-1260	11096-82-5			0.001	0.00031	0.000022
Aroclor-1262	37324-23-5			0.001	0.00031	0.000047
Aroclor-1268	11100-14-4			0.001	0.00031	0.000053
Total PCBs	N/A	0.01	CQAP	0.001	0.00031	0.0001

Note:
a - Achievable QLs and DLs are limits for example purposes and may vary based on the laboratory selected to conduct the analyses. These QLs and DLs are also what the laboratory may be able to achieve when performing the analytical methods specified in Worksheet #23 with nominal sample volumes in the absence of interferences. Actual DLs and QLs will vary based on sample specific factors. Should widespread QL exceedances occur for one or more analytical groups, corrective actions will include alternative cleanups, reanalyses at lesser dilutions, and/or repreparations/re-extractions. Matrix interferences are not always overcome by corrective actions and laboratory QLs may be elevated in these instances. The project chemist will work with the laboratory to propose alternative analytical approaches, if available, to overcome the exceedances.

Acronyms:
CAS - Chemical Abstracts Service
CQAP - Construction Quality Assurance Plan
DL - Detection Limit
N/A - Not applicable
PAL - Project Action Limit
QL - Quantitation Limit
µg/m^3 - micrograms per cubic meter

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

QAPP Worksheets #15A and #15D: Sediment/Soil

Analyte	CAS Number	PAL	PAL Reference	Project Quantitation Goal	Achievable Laboratory QLs[1]	Achievable Laboratory DLs[1]
Geotechnical						
GAC Content (%)	N/A	N/A	N/A	N/A	N/A	N/A

Notes:

1. Achievable QLs and DLs are limits for example purposes and may vary based on the laboratory selected to conduct the analyses. These QLs and DLs are also what the laboratory may be able to achieve when performing the analytical methods specified in Worksheet #23 with nominal sample volumes in the absence of interferences. Actual DLs and QLs will vary based on sample-specific factors. Should widespread QL exceedances occur for one or more analytical groups, corrective actions will include alternative cleanups, re-analyses at lesser dilutions, and repreparations or re-extractions. Matrix interferences are not always overcome by corrective actions, and laboratory QLs may be elevated in these instances. The project chemist will work with the laboratory to propose alternative analytical approaches, if available, to overcome the exceedances.

Acronyms:
CAS: Chemical Abstracts Service
DL: detection limit
GAC: granular activated carbon
N/A: not applicable
PAL: project action limit
QL: quantitation limit

QAPP Worksheet #17: Sampling Design and Rationale

QAPP Worksheet #17C1: Task C1 - Post-Dredge/Excavation Sediment and Soil Sampling

TASK C1: SEDIMENT CHARACTERIZATION

Following sediment and soil removal, post-dredge/excavation sampling will be conducted to verify that removal has addressed PCB contamination in the designated areas as outlined in the Construction Quality Assurance Plan (CQAP; Arconic, June 2019).

Target locations may be adjusted in the field based on conditions encountered and professional judgment of the field crew conducting the efforts. Core segmentation may also be altered in the field to account for changes in stratigraphy.

Per the April 2013 ROD, the near shore areas are defined as the submerged area between the upland and the location where the gentle bathymetric slope along the shoreline meets the steep slope of the main channel side walls (generally these areas have 5 feet or less of water during normal summer flow and extend approximately 25 feet from shore). Sampling density is required at a rate of 8 cores per acre.

Sediment samples will be submitted for total polychlorinated biphenyls (PCBs) (Aroclors) and total solids, as discussed on Worksheet #11C1. In addition, field data including water surface elevation, water depth, sediment descriptions and sediment probing, penetration, and recovery depths will be recorded.

Post-Dredge/Excavation Sediment and Soil Sampling will begin in spring 2019 and is expected to continue through fall 2019. Sampling activities will be conducted consistent with the field sampling SOPs provided in the CQAP.

QAPP Worksheet #17: Sampling Design and Rationale

QAPP Worksheet #17C2: Task C2 - Water Quality Monitoring

TASK C2: Water Quality Monitoring

During in-river and adjacent floodplain debris removal, dredging, excavation, backfilling, and capping, water column and water intake monitoring will be performed, as outlined in the Construction Quality Assurance Plan (CQAP; Arconic, June 2019), to assess the potential impacts of remedial activities.

Locations for water column monitoring include a fixed location upstream (upstream of remedial action areas), a near-field station (mobile station approximately 1,000 feet downstream of the work areas), a fixed location established at the historic water column sampling location WC013 (located just upstream of the Grasse River mouth at T71), and a far-field station in the St. Lawrence River. The Arconic East Plant and St. Regis Mohawk Tribe water intakes will also be sampled at the ports within each facility.

Daily samples will be submitted for PCB Aroclor and TSS analysis from spring 2019 through fall 2019, as discussed on Worksheet #11C2. In addition, grab samples will be collected once a week for the first month of near shore dredging, debris removal, and backfilling from the near-field station and submitted for analysis of lead, mercury, and select polycyclic aromatic hydrocarbons (PAHs).

Sampling activities will be conducted consistent with the field sampling SOPs provided in the CQAP.

QAPP Worksheet #18: Sampling Locations and Methods

Sample Location/ID Number[1,2]	Matrix	Depth (units)[3,4]	Analytical Group	Type	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
332 Locations	Sediment	6 inch increments to end of recovered material (see note 3)	PCBs and total solids	Vibracore or manual coring	Estimated field samples = 996 Field duplicates (collected at a rate of 1 per 20 field samples) = 50 samples Total samples = 1,046	EMP, SOP 1, 5	Verification of the removal of PCB contaminated sediment
38 Locations	Soil	0 to 6 inches	PCBs and total solids	Push core	Estimated field samples = 38 Field duplicates (collected at a rate of 1 per 20 field samples) = 2 samples Total samples = 40	EMP, SOP 2, 5	Verification of the removal of PCB contaminated soil
4 In-river Locations (WC-UP, WC-NF, WC-MOUTH, WC-SLR)	Water	0.2 and 0.8 times the water depth; If water depth is < 5 feet then 0.5 times water depth	PCBs (excluding WC-UP) and TSS	Kemmerer water sampler	Estimated field samples = ~800 Field duplicates (collected at a rate of 1 per 20 field samples) = ~40 sample Total samples = ~840	EMP, SOP 3, 5	Monitor water quality impacts resulting from remedial activities
2 Intake Locations (Arconic East Plant and SRMT intakes)		N/A	PCBs	Grab	Estimated field samples = ~600 Field duplicates (collected at a rate of 1 per 20 field samples) = ~30 sample Total samples = ~630		
WC-NF		0.2 and 0.8 times the water depth	PAHs, Metals	Kemmerer water sampler	Estimated field samples = 4 Field duplicates (collected at a rate of 1 per 20 field samples) = 1 sample Total samples = 5		

Notes:

1. Post-dredge sampling locations are shown on Attachment B of the CQAP. Water quality sampling locations are shown in the EMP (Attachment D to the CQAP).
2. WC-UP will only be sampled for TSS (i.e., PCB samples will not be collected from this location).
3. All sections down to the top 18 inches (or top of native sediments) will be submitted for analysis with the remaining segments archived pending the results of the upper segments. Additional sediment/soil sampling may be conducted if additional dredge/excavation passes are required.
4. In-river water column samples will be composites of samples collected from 0.2 and 0.8 times the water depth, unless stratification is observed.

Acronyms:
CQAP: Construction Quality Assurance Project Plan
EMP: Environmental Monitoring Plan
N/A: not applicable
PAH: polycyclic aromatic hydrocarbon
PCB: polychlorinated biphenyl
SOP: standard operating procedure
TSS: total suspended solids

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times

Laboratory (Name, sample receipt address, point of contact, e-mail, and phone numbers):
Chemical Analyses (PCB, total solids, and TSS) – Pace Analytical Services, LLC, Mobile Laboratory; 2525 Advance Road, Madison, WI 53718; Pat Letterer; Pat.Letterer@pacelabs.com; (608) 221-8700
List any required accreditations/certifications: Pace Analytical – NY DOH ELAP
Back-up Laboratory: Eurofins Lancaster Laboratories Environmental, 2425 New Holland Pike, Lancaster, PA 17605; (717) 656-2300
Sample Delivery Method: Courier/Commercial Shipping Company (if samples are shipped to the Back-up laboratory)

Analyte/ Analyte Group	Matrix	Method/ SOP	Accreditation Expiration Date[1]	Container(s) (number, size & type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Turnaround[3]
PCB (Aroclor)	Sediment/ Soil	SW-846 8082/ P-07, P-23, E-07	April 22, 2020 (April 1, 2020)	1, 4-oz. glass jar	4 ± 2°C or -18 ± 10°C[2]	None	1 year to extraction; 40 days from extraction to extract analysis	24 hours
Total Solids	Sediment/ Soil	SM2540B/ P-24, E-09	April 22, 2020 (April 1, 2020)	1, 4-oz. glass jar	4 ± 2°C or -18 ± 10°C[2]	N/A	14 days if stored refrigerated, 6 months if stored frozen	24 hours
PCB (Aroclor)	Water	SW-846 8082/ P-07, P-22, E-06	April 22, 2020 (April 1, 2020)	1L, glass amber bottle	4 ± 2°C	None	None	24 hours
TSS	Water	SM2540D/ P-25, E-10	April 22, 2020 (April 1, 2020)	1L, plastic bottle	4 ± 2°C	N/A	7 days	24 hours
PCB (Congener)	Water	EPA 1668A/E-18	April 1, 2020	1L, glass amber bottle	4 ± 2°C	None	1 year to extraction; 1 year from extraction to extract analysis	14 calendar days
PAHs	Water	SW-846-8270D-SIM/E-11, E-12	April 1, 2020	125 mL, glass amber bottle	4 ± 2°C	7 days	40 days	24 hours
Metals	Water	SW-846-6020A, SW-846-7470A/E-13, E-14, E-15, E-16	April 1, 2020	125 mL plastic bottle	4 ± 2°C, HNO3	6 months; 28 days for Hg	6 months; 28 days for Hg	24 hours

Notes:
1. Accreditation expiration date for Pace Analytical Services, LLC, Mobile Laboratory is approximate. The expiration date for the back-up laboratory is provided in parentheses.
2. In-process samples will be analyzed immediately in the mobile lab or refrigerated at 4 ± 2°C until extraction. Archived sediment samples will be frozen upon receipt at the laboratory.
3. Data to be provided as an electronic data deliverable (EDD) within the specified turnaround, with laboratory reports provided subsequently.

Acronyms:
C: Celsius
Hg: mercury
L: liter
mL: milliliter
NY DOH ELAP: New York Department of Health Environmental Laboratory Accreditation Program
N/A: not applicable
oz: ounce
PCB: polychlorinated biphenyl
SOP: standard operating procedure
TSS: total suspended solids

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times

Laboratory (Name, sample receipt address, point of contact, e-mail, and phone numbers):
Chemical Analyses (PCB) – TestAmerica Burlington; 30 Community Drive, South Burlington, VT, 05403; Kathryn Kelly; kathryn.kelly@testamericainc.com; 802-923-1021
List any required accreditations/certifications: NY DOH ELAP
Sample Delivery Method: Courier/Commercial Shipping Company

Analyte/ Analyte Group	Matrix	Method/ SOP	Accreditation Expiration Date	Container(s) (number, size & type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
PCB (Aroclor)	Air	SW846-8082A, USEPA TO4A/TO10A /BR-GC-005	4/1/2019	Pre-cleaned filters and cartridges	Stored protected from light in a resealable bag in a jar; exposed cartridges will be returned to the jar and maintained at a temperature of 4°C ± 2°C	None	None	30 calendar days

Acronyms:
C: Celsius
NY DOH ELAP: New York Department of Health Environmental Laboratory Accreditation Program
PCB: polychlorinated biphenyl
SOP: standard operating procedure

QAPP Worksheet #19 and 30: Sample Containers, Preservation, and Hold Times

Laboratory (Name, sample receipt address, point of contact, e-mail, and phone numbers):
Thermal burn: Atlantic Testing Laboratories (ATL), Limited; 6085 Court Street Road, Suite A, Syracuse, New York 13206; Daniel Owens; dowens@atlantictesting.com; (315) 699-5281
Heavy liquid separation: Anchor QEA Engineering, PLLC; 6720 SW Macadam Avenue Suite 125, Portland, Oregon 97219; Jessica Goin; jgoin@anchorqea.com; (503) 972-5019
List any required accreditations/certifications: N/A
Backup Laboratory: N/A
Sample Delivery Method: Courier/Commercial Shipping Company

Analyte/ Analyte Group	Matrix	Method/SOP	Accreditation Expiration Date	Container(s) (number, size, and type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
GAC Content (thermal burn)	Sediment/Soil	SOP 4	N/A	1, 2-kg bucket	None	N/A	None	30 calendar days
GAC Content (heavy liquid separation)	Sediment/Soil	SOP 4 Attachment 2	N/A	1, 4-oz jar	None	N/A	None	30 calendar days

Acronyms:
GAC: granular activated carbon
kg: kilogram
N/A: not applicable
oz: ounce
SOP: standard operating procedure

QAPP Worksheet #20: Field QC Summary

Matrix	Analyte/ Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Duplicates /Matrix Spike Duplicates	Field Blanks	Equipment Blanks	Trip Blanks	Other	Total # Analyses
Sediment and Soil	PCB (Aroclor)	~996 Sediment; 38 Soil	1 per 20 collected	1 per 20 collected	1 per 20 collected	None	1 each sampling day start, and 1 at conclusion of all sampling activities if using non-disposable equipment	None	N/A	1,146 Sediment; 44 Soil
	Total Solids			N/A		N/A	N/A	N/A	N/A	1,046
Water	PCB (Aroclor)	~800	1 per 20 collected	1 per 20 collected	1 per 20 collected	None	1 each sampling day start and 1 at conclusion of all sampling activities	None	N/A	~920
	TSS	~600		N/A		N/A	N/A	N/A	N/A	~660
	PCB (Congener)	8	1 per 20 collected	N/A	1 per 20 collected	N/A	None	None	N/A	11
	PAHs	4	1	1	1	None	1	N/A	N/A	8
	Metals	4	1	1	1	N/A	1	N/A	N/A	8
Air	PCB (Aroclor)	~1,500	1 per 20 collected	None	None	None	None	None	N/A	~1,800

QAPP Worksheet #20: Field QC Summary (Cont'd)

Matrix	Analyte/ Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Duplicates /Matrix Spike Duplicates	Field Blanks	Equipment Blanks	Trip Blanks	Other	Total # Analyses
Sediment/ Soil	GAC Content	1 ex situ sample per 5,000 tons of chemical isolation material; 1 composite in situ sample per test cell (6 total)	None	None	None	None	None	None	N/A	All field samples will be run in triplicate for both methods; ~24 total per method; ~48 total analyses

Notes:
1. Additional sediment/soil sampling may be conducted if additional dredge/excavation passes are required.
2. During the Staged Capping Test, ex situ samples will be collected daily from the sand only stockpile to test for naturally occurring organics in the source material used in the chemical isolation layer and post-mixing (i.e., sand and GAC).

Acronyms:
GAC: granular activated carbon
N/A: tot applicable
PAH: polycyclic aromatic hydrocarbon
PCB: polychlorinated biphenyl
TSS: total suspended solids

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment	Activity	SOP Reference	Title or Position of Responsible Person	Frequency	Acceptance Criteria	Corrective Action
Real-time air meters	Calibration, Maintenance, Testing, and Inspection	Operations Manual	Field technician	In accordance with Operations Manual	In accordance with Operations Manual	In accordance with Operations Manual
Low or high volume air samplers	Calibration, Maintenance, Testing, and Inspection	Operations Manual	Field technician	In accordance with Operations Manual	In accordance with Operations Manual	In accordance with Operations Manual

Acronym:
SOP: standard operating procedure

QAPP Worksheet #23: Analytical SOPs

SOP #[1]	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix/Analytical Group	Instrument/ Equipment	Organization Performing Analysis	Modified for Project Work? (Y/N)[2]
P-07	*Determination of Polychlorinated Biphenyls (PCBs) Aroclors; Rev.03a; 09/27/2016; S-NY-O-314*	Definitive	Sediment, Aqueous, Tissue/Organics	GC/ECD	Pace Analytical Services, Inc.	Y
P-20	*Chain-of-Custody, Log-In, Tracking Procedures and Sample Containers; Rev. 4.9; 02/02/2016; GEN-003*	N/A	N/A	Thermometer	Pace Analytical Services, Inc.	N
P-21	*Waste Handling and Management; Rev. 01; 03/31/2017; MAD-W-002*	N/A	N/A	None	Pace Analytical Services, Inc.	N
P-22	*Self-Contained Water Extraction; Rev.0.4; 03/28/2016; PRE-002*	Definitive	Aqueous/Organics (Sample Extraction)	Sample glassware, measurement syringes, etc. and use of solvent/reagents	Pace Analytical Services, Inc.	N
P-23	*Self-Contained Soil Extraction; Rev.0.4; 03/28/2016; PRE-003*	Definitive	Sediment/Organics (Sample Extraction)	Analytical balance, sample glassware measurement syringes, etc. and use of solvent/reagents	Pace Analytical Services, Inc.	N
P-24	*Determination of Total Solids; Rev.5.4; 02/02/2016; GEN-007*	Definitive	Sediment/General Chemistry	Analytical Balance	Pace Analytical Services, Inc.	N
P-25	*Determination of Total Suspended Solids; Rev.3.6; 02/02/2016; LAM-012*	Definitive	Aqueous/General Chemistry	Analytical Balance	Pace Analytical Services, Inc.	N
E-01	*Environmental Sample Receipt and Unpacking; Rev. 17; 11/02/2018; S-SA-WI10725*	N/A	N/A	Thermometer	Eurofins Lancaster Laboratories Environmental	N
E-02	*Automated Storage, Retrieval, and Discarding of Samples; Rev. 10; 03/15/2017; S-SS-WI12042*	N/A	N/A	N/A	Eurofins Lancaster Laboratories Environmental	N
E-03	*Laboratory Sample Analysis Record (LSAR) Documentation; Rev. 8; 05/04/2018; QA-SOP11184*	N/A	N/A	N/A	Eurofins Lancaster Laboratories Environmental	N
E-04	*Management of Hazardous Wastes in the Laboratory; Rev. 4; 09/04/2018; G-EHS-SOP22000*	N/A	N/A	None	Eurofins Lancaster Laboratories Environmental	N

QAPP Worksheet #23: Analytical SOPs (Cont'd)

SOP #[1]	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix/Analytical Group	Instrument/ Equipment	Organization Performing Analysis	Modified for Project Work? (Y/N)[2]
E-05	*Microwave Extraction Method 3546 for PCBs in a Solid Matrix; Rev. 8; 05/01/2018; T-OE-PEST-WI10927*	Definitive	Sediment/Organics (Sample Preparation)	MARS Xpress Microwave Extractor	Eurofins Lancaster Laboratories Environmental	N
E-06	*Separatory Funnel Extraction by Method 3510C, 608 or 622 for Pesticides and PCBs in a Wastewater; Rev. 19; 10/07/2016; T-OE-PEST-WI10920*	Definitive	Aqueous/Organics (Sample Preparation)	Separatory Funnel	Eurofins Lancaster Laboratories Environmental	N
E-07	*Polychlorinated Biphenyls (PCBs) in Solid Samples by 8082A Using GC-ECD; Rev. 9; 03/29/2018; T-PEST-WI10004*	Definitive	Sediment, Organics	GC/ECD	Eurofins Lancaster Laboratories Environmental	Y
E-08	*Analysis of Polychlorinated Biphenyls (PCBs) by 8082A in Aqueous Samples Using GC-ECD; Rev. 6; 03/29/2018; T-PEST-WI9238*	Definitive	Aqueous/Organics	GC/ECD	Eurofins Lancaster Laboratories Environmental	Y
E-09	*% Moisture Calculation (Gravimetric) by SM 2540 G-1997; Rev. 16; 12/28/2017; S-SS-WI10697*	Definitive	Sediment/General Chemistry	Analytical balance	Eurofins Lancaster Laboratories Environmental	Y
E-10	*Total Suspended Solids (TSS) - Gravimetric by SM 2540 D2011 or SM 2540 D1997 and Total Filtered: Total Volume Test by NJDEP in Waters; Rev. 11; 12/24/2018; T-WC-WI11604*	Definitive	Aqueous/General Chemistry	Analytical Balance	Eurofins Lancaster Laboratories Environmental	N
E-11	*Separatory Funnel Extraction by Method 3510C for BNAs by 8270 SIM in Wastewater; Rev. 9; 03/20/2019; T-OE-SVOA-WI10931*	Definitive	Aqueous/Organics (Sample Preparation)	Separatory funnel	Eurofins Lancaster Laboratories Environmental	N
E-12	*Semivolatiles in Waters and Soils by Methods 8270C/D SIM by GC/MS; Rev. 14; 03/21/2019; T-SVOA-WI9995*	Definitive	Sediment, Aqueous/Organics	GC/MS	Eurofins Lancaster Laboratories Environmental	N

QAPP Worksheet #23: Analytical SOPs (Cont'd)

SOP #[1]	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix/Analytical Group	Instrument/ Equipment	Organization Performing Analysis	Modified for Project Work? (Y/N)[2]
E-13	*Sample Preparation of Leachates and Other Wastewater for Analysis of Total Metals by Inductively Coupled Plasma-Mass Spectrometer (ICPMS); Rev. 12; 01/19/2015; T-MET-WI11937*	Definitive	Aqueous/Metals (Sample Preparation)	Hotblock/hotplate	Eurofins Lancaster Laboratories Environmental	N
E-14	*Metals by Inductively Coupled Plasma Mass Spectrometry for SW846 Methods 6020/6020A/6020B (aqueous, solid, tissue) and EPA 200.8 (aqueous); Rev. 9; 03/21/2019; T-MET-WI11933*	Definitive	Sediment, Aqueous, Tissue/Metals	ICP/MS	Eurofins Lancaster Laboratories Environmental	N
E-15	*Digestion of Aqueous Samples by SW846 Method 7470A, EPA 245.1; Rev. 20; 03/21/2019; T-MET-WI11924*	Definitive	Aqueous/Metals	Hotblock	Eurofins Lancaster Laboratories Environmental	N
E-16	*Mercury in Aqueous, Solid and Tissue Samples by EPA 7471A, 7471B, 7470A, and 245.1 re/v 3 by Cold Vapor AA; Rev. 17; 03/08/2019; T-MET-WI7965*	Definitive	Sediment, Aqueous, Tissue/Metals	CVAAS	Eurofins Lancaster Laboratories Environmental	N
E-17	*Separatory Funnel Extraction Procedure for HRMS Analysis in an Aqueous Matrix Using Method 1613B, 8290A, 1668A, and 1668C; Rev. 4; 12/05/2018; T-HRMS-WI12032*	Definitive	Sediment, Aqueous, Tissue/Organics	Separatory Funnel	Eurofins Lancaster Laboratories Environmental	N
E-18	*PCB Congeners by Method 1668 HRGC/HRMS in Aqueous and Solid Matrices; Rev. 7; 05/30/2018; T-HRMS-WI9432*	Definitive	Sediment, Aqueous, Tissue/Organics	HRGC/HRMS	Eurofins Lancaster Laboratories Environmental	Y
BR-GC-005	*Polychlorinated Biphenyls (PCBs) by GC/ECD (SW846 8082A, USEPA TO4A/TO10A; Rev. 13.0, 01/24/18*	Definitive	Air/Organics	GC/ECD	TestAmerica, Inc.	N

QAPP Worksheet #23: Analytical SOPs (Cont'd)

SOP #[1]	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix/Analytical Group	Instrument/ Equipment	Organization Performing Analysis	Modified for Project Work? (Y/N)[2]
BR-EX-025	*Media Prep and Extraction Procedure for PCBs in Ambient Air by SW-846 3540 and 3541 (EPA TO4/TO10); Rev. 2.1, 08/28/18*	Definitive	Air/Organics	Soxhlet	TestAmerica, Inc.	N

Notes:
1. All Analytical SOPs are attached at the end of the worksheets.
2. Modifications to SOPs are based on project-specific criteria outlined in Worksheet #28.

Acronyms:
CVAAS: cold vapor atomic absorption spectrometer
ECD: electron capture detector
HR: high resolution
MS: mass spectrometer
N/A: not applicable
GC: gas chromatograph
SOP: standard operating procedure
ICP: Inductively coupled plasma
TSS: total suspended solids

QAPP Worksheet #24: Analytical Instrument Calibration

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference[1]
GC/ECD (PCB-Aroclor and Congeners)	Initial and continuing calibration as required in SOP	See SOP	ICAL after instrument set up, after major instrument changes and when continuing calibration criteria are not met. Continuing calibration every 12 hours or after 20 samples.	ICAL %RSD $\leq$ 20% or linear curve r$\geq$ 0.995 or r^2 $\geq$0.990, or quadratic curve r^2 $\geq$0.990. CCV %D $\leq$ 15%	Inspect system, correct problem, rerun calibration and affected samples	Analyst	P-07, E-07, and E-08
GC/MS (PAHs)	Decafluorotriphenyl phosphine tune; initial and continuing calibration as required in SOP	See SOP	Verify tune and CCV every 12 hours; ICAL after instrument set up, after major maintenance, and/or after instrument changes have occurred	ICAL %RSD ≤20%; no more than 10% or alternative curve fits ICV ≤30% D CCV %D ≤20%	Inspect system, correct problem, rerun calibration and affected samples	Analyst	E-12
ICP/MS (metals)	Daily tune and performance check; initial and continuing calibration per SOP	See SOP	Performance check daily; ICAL daily or per batch; CCV every ten samples	Tuning solution analytes % RSD ≤ 5%; mass calibration ± 0.1 AMU; resolution < 0.9 AMU at 10% peak height ICAL r ≥0.995; ICV, CCV ±10%	Inspect system, correct problem, rerun calibration and affected samples	Analyst	E-14
CVAAS (mercury)	Initial and continuing calibration per SOP	See SOP	ICAL daily; CCV every ten samples	ICAL r ≥0.995; ICV ± 10%; CCV ± 20%	Inspect system, correct problem, rerun calibration and affected samples	Analyst	E-16
HRGC/ HRMS (PCB Congeners)	Perfluorokerosene tune, initial and continuing calibration as required in SOP	See SOP	ICAL after instrument set up, after major instrument changes, and when continuing calibration criteria are not met; calibration verification minimum every 12 hours	ICAL %RSD <20% for target analytes calculated by isotope dilution %RSD <35% for target analytes calculated by internal standard CCV %D ±30% for unlabeled compounds, ±50% for labeled compounds	Inspect system, correct problem, rerun calibration and affected samples	Analyst	E-18
Analytical Balance (Total Solids, TSS)	Daily	See SOP	Weigh and record NIST traceable standard weight in range of interest	± 5% of certified weight	Inspect system, correct problem, rerun calibration and affected samples	Analyst	P-24, P-25, E-09, and E-10

QAPP Worksheet #24: Analytical Instrument Calibration (Cont'd)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference[1]
GC/ECD (PCB-Aroclors)	Initial and continuing calibration as required in SOP	See SOP	ICAL after instrument set up, after major instrument changes and when continuing calibration criteria are not met. Continuing calibration every 12 hours or after 20 samples, whichever is more frequent.	ICAL %RSD $\leq$ 20% or linear curve r$\geq$ 0.995 or r^2 $\geq$0.990, or quadratic curve r^2 $\geq$0.990. ICV %R ± 20%; CCV %D $\leq$ 20%	Inspect system, correct problem, rerun calibration and affected samples	Laboratory Manager	BR-GC-005

Note:
1. Refer to the Analytical SOP Reference Table (Worksheet #23).

Acronyms:
%D: percent difference or percent drift
%R: percent recovery
CCV: continuing calibration verification
CVAAS: cold vapor atomic absorption spectrophotometer
ECD: electron capture detector
g: grams
GC: gas chromatograph
HR: high resolution
ICAL: initial calibration
MS: mass spectrometer
N/A: not applicable
NIST: National Institute of Standards and Technology
PAH: polycyclic aromatic hydrocarbon
PCB: polychlorinated biphenyl
RSD: relative standard deviation
SOP: standard operating procedure
TSS: total suspended solids

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference[1]
GC/ECD (PCB Aroclors and Congeners)	Change septa, clean injectors, change or trim columns, install new liners	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	See SOP	See SOP	Laboratory Manager	P-07, E-07, and E-08
GC/MS (PAHs)	Change septa; clean injectors; change or trim columns; install new liners; clean sources and quadruple rods; maintain vacuum pumps	Tuning	Instrument performance and sensitivity	As needed	See SOP	See SOP	Analyst or section supervisor	E-12
ICP/MS (metals)	Clean cones; check pump tubing; flush lines; clean aspiration chamber and ion lens	Tuning	Check connections	Daily or as needed	See SOP	See SOP	Analyst or section supervisor	E-14
CVAAS (mercury)	Replace tubing, clean optical cell and windows	Sensitivity check	Check connections	Daily or as needed	See SOP	See SOP	Analyst or department supervisor	E-16
HRGC/HRMS (PCB congeners)	Change septa; clean injectors; change or trim columns; install new liners; clean sources and quadruple rods; maintain vacuum pumps	Tuning	Instrument performance and sensitivity	As needed	See SOP	See SOP	Analyst or department supervisor	E-18
Analytical Balance (Total Solids, TSS)	Clean balance after each use; service annually	NIST Traceable weights	Balance performance and sensitivity	Prior to every use	Measured weight within certified tolerance	Clean, verify zero on balance, reweigh; call for service	Laboratory Manager	P-24, P-25, E-09, and E-10

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection (Cont'd)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/Position Responsible for Corrective Action	SOP Reference[1]
GC/ECD (PCB Aroclors)	Change septa, clean injectors, change or trim columns, install new liners	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	See SOP	See SOP	Laboratory Manager	BR-GC-005

Note:
1. Refer to the Analytical SOP Reference Table (Worksheet #23).

Acronyms:
CVAAS: cold vapor atomic absorption spectrophotometer
ECD: electron capture detector
g: grams
GC: gas chromatograph
HR: high resolution
MS: mass spectrometer
NIST: National Institute of Standards and Technology
PAH: polycyclic aromatic hydrocarbon
PCB: polychlorinated biphenyl
SOP: standard operating procedure
TSS: total suspended solids

QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal

Sampling Organization: ARCADIS

Laboratory: TestAmerica Burlington

Method of sample delivery (shipper/carrier): Courier/Commercial Shipping Company

Number of days from reporting until sample disposal: Varies[1]

Activity	Organization and Title or Position of Person Responsible for the Activity	SOP Reference
Sample labeling	ARCADIS	PDI QAPP (October 2013), 6
Chain-of-custody form completion	ARCADIS	PDI QAPP (October 2013), 6
Packaging	ARCADIS	PDI QAPP (October 2013), 6
Shipping coordination	ARCADIS	PDI QAPP (October 2013), 6
Sample receipt, inspection, & log-in	TestAmerica	BR-EX-025
Sample custody and storage	TestAmerica	BR-EX-025
Sample disposal	TestAmerica	BR-EX-025

Note:

1. Samples will be stored at the laboratory as directed. No samples will be disposed of without prior approval by the Project Manager.

Acronyms:

PDI QAPP: Pre-design Investigation Quality Assurance Project Plan

SOP: standard operating procedure

QAPP Worksheet #26 and 27: Sample Handling, Custody, and Disposal

Sampling Organization: Anchor QEA Engineering, PLLC

Laboratory: Atlantic Testing Laboratories, Limited, and Anchor QEA Engineering, PLLC

Method of sample delivery (shipper/carrier): Courier/Commercial Shipping Company

Number of days from reporting until sample disposal: Varies[1]

Activity	Organization and Title or Position of Person Responsible for the Activity[2]	SOP Reference
Sample labeling	Anchor QEA	4
Chain-of-custody form completion	Anchor QEA	4
Packaging	Anchor QEA	4
Shipping coordination	Anchor QEA	4
Sample receipt, inspection, and log-in	Atlantic Testing Laboratories/Anchor QEA	4
Sample custody and storage	Atlantic Testing Laboratories/Anchor QEA	4
Sample disposal	Atlantic Testing Laboratories/Anchor QEA	4

Notes:
1. Samples will be stored at the laboratory as directed. No samples will be disposed of without prior approval by the Arconic Project Manager.
2. During the Staged Capping Test, granular activated carbon samples will be shipped to and analyzed by Atlantic Testing Laboratories and Anchor QEA.

Acronym:
SOP: standard operating procedure

QAPP Worksheet #28: Analytical Quality Control and Corrective Action

Matrix Air
Analytical Group and Method PCB Aroclors (TO4A and TO10A)
Analytical SOP BR-GC-005

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method blank	1 per batch (up to 20 samples)	No Target Compounds >QL	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report results if sample results >5x blank result or sample results ND. If contamination is widespread or reoccurring, analyses must stop and the source of contamination must be eliminated or reduced before analyses can continue.	Laboratory Manager, Quality Manager	No Target Compounds >QL
Surrogates	Every sample	60%-120% R	Check calculations and instrument performance; recalculate, reanalyze. Qualify data as needed.	Laboratory Manager, Quality Manager	%R: 60%-120%
Laboratory control sample	1 per batch (up to 20 samples)	65%-125% R	Reprepare and/or reanalyze affected samples. Qualify data as needed.	Laboratory Manager, Quality Manager	%R: 65%-125%
Laboratory control sample duplicate	1 per batch (up to 20 samples)	65%-125% R; RPD ≤30%	Reprepare and/or reanalyze affected samples. Qualify data as needed.	Laboratory Manager, Quality Manager	%R: 65%-125%; RPD ≤30%
Field Duplicate	1 per 20 field samples	RPD ≤50% if both results are >5x QL. Difference ≤ QL if results are ≤5x QL	Evaluate during data validation.	Quality Manager	RPD ≤50% if both samples are >5x QL. Difference ≤ QL if results are ≤5x QL.

Acronyms:
QC: quality control
QL: quantitation limit
%R: percent recovery
RPD: relative percent difference
SOP: standard operating procedure

QAPP Worksheet #28: Analytical Quality Control and Corrective Action

Matrix	Sediment/Soil
Analytical Group and Method	GAC Content (Thermal burn and Heavy Liquid Separation)
Analytical SOP	SOP 4

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Laboratory control sample duplicate	None	N/A	N/A	N/A	N/A
Field Duplicate	None	N/A	N/A	N/A	N/A

Note:
1. Atlantic Testing Laboratories and Anchor QEA will run triplicate analysis on the GAC samples during the Staged Capping Test.

Acronyms:
GAC: granular activated carbon
N/A: not applicable
QC: quality control
SOP: standard operating procedure

ENVIRONMENTAL MONITORING PLAN GRASSE RIVER

Prepared for

Arconic, Inc.

June 2019

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LIST OF ACRONYMS AND ABBREVIATIONS

6 NYCRR	Title 6 of the New York Codes, Rules and Regulations
µg/L	microgram per liter
Arconic	Arconic, Inc.
BMP	best management practice
CQAP	Construction Quality Assurance Plan
DQO	data quality objective
EMP	Environmental Monitoring Plan
FDR	Final Design Report
L/min	liter per minute
mg/L	milligram per liter
mg/m^3	milligram per cubic meter
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PDI	Pre-Design Investigation
PM_{10}	particulate matter less than 10 microns
ppm	part per million
PUF	polyurethane foam
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
ROD	*Record of Decision*
ROPS	Remedial Options Pilot Study
RSI	River and Sediment Investigation
SLF	secure landfill
SOP	standard operating procedure

SRMT	Saint Regis Mohawk Tribe
TAT	turn-around-time
TOGS	Technical and Operational Guidance Series
TSS	total suspended solids
USEPA	U.S. Environmental Protection Agency
VOC	volatile organic compound

1 INTRODUCTION

This Environmental Monitoring Plan (EMP) has been prepared to outline the monitoring activities that Arconic, Inc. (Arconic), will conduct during the remedial construction work to be performed in the Lower Grasse River. As described in the Final Design Report (FDR), remedial activities to meet the requirements of the Grasse River Superfund Site *Record of Decision* (ROD; U. S. Environmental Protection Agency [USEPA], April 2013) will include near shore dredging and backfilling, select floodplain soil removal and backfilling, cap placement in the main channel, restoration, and associated support activities such as material processing and landfilling. Monitoring will be performed during these activities to assess the potential impacts of remediation on water and air quality.

This EMP is specific to environmental monitoring to be performed during active remediation. Ice monitoring will also be performed each year during the remediation project winter shutdown period in accordance with the program presented and discussed at the USEPA Technical Team meeting on December 18, 2008 and January 2009 Ice Monitoring Scope (Appendix A) and will continue through the end of remediation. Construction-related operations monitoring activities to be conducted during remediation are provided in the main body of the Construction Quality Assurance Plan (CQAP). Long-term monitoring efforts (i.e., post-remediation) will be specified in the Operations, Maintenance, and Monitoring Plan (Appendix J to the FDR), and the scope of monitoring for habitat recovery will be provided in the Habitat Reconstruction Plan (provided under separate cover).

This EMP is an attachment to the CQAP, which is Appendix C to the FDR. As such, background information on the site and associated characteristics and details on the remedial activities are not repeated herein but can be found in the main bodies of the CQAP and FDR.

Details regarding the environmental monitoring for the water column and air are provided in Sections 2 and 3, respectively. Section 4 outlines the quality assurance (QA) and quality control (QC) for the environmental monitoring program, and Section 5 provides the references for this EMP. The remainder of this section provides the data quality objectives (DQOs) for the environmental monitoring program.

1.1 Data Quality Objectives

The overall objective of the environmental monitoring program is to obtain data that can be used to assess the need for corrective actions during construction. DQOs specific to this environmental monitoring program were developed to guide the monitoring components to ensure the data collected would be appropriate for their intended use during remediation. DQOs have been developed for each of the media listed in this section.

The following DQOs have been established for water:

- Assess solids levels upstream and downstream of near shore dredging, debris removal, backfilling, and capping activities to determine whether corrective actions are needed.
- Assess downstream impacts of near shore dredging, debris removal, and backfilling activities on polychlorinated biphenyl (PCB) levels to determine whether corrective actions are needed.

The following DQOs have been established for air:

- Assess particulate and volatile organic compound (VOC) levels in ambient air conditions upwind and downwind of near shore dredging, sediment processing, landfilling, and backfill and cap material management activities to determine whether corrective actions are needed to address emissions that could potentially affect off-site receptors (i.e., locations such as residences or businesses where individuals not involved with the remedial activities may be present for a reasonable period of time)
- Assess airborne releases of PCBs downwind of near shore dredging, sediment processing, and landfilling activities to determine whether corrective actions are needed to address emissions that could potentially affect off-site receptors

2 WATER MONITORING

Water monitoring will include water column and water intake monitoring during all intrusive activities with the potential to impact the river including near shore dredging, debris removal, floodplain soil removal, backfilling, and capping activities. Based on Grasse River site-specific experiences during the 2005 *Remedial Options Pilot Study* (ROPS; Alcoa, May 2006) and the 2001 *Capping Pilot Study* (Alcoa, April 2002), along with other sediment remediation projects, sediment resuspension during dredging and associated activities is likely, but it is not expected during capping activities. Therefore, water monitoring for PCBs will be initiated at the start of the in-water near shore dredging and debris removal activities and is anticipated to continue through completion of near shore backfilling. A reduced water monitoring program will be conducted during capping activities. The following sections outline the monitoring program for the water column and water intakes, as well as the associated advisory and corrective action triggers. The advisory levels will inform construction operations and adjustments, and corrective action levels determine potential community exposure and the need for corrective actions.

Laboratory analyses will be performed by a laboratory certified by the New York State Department of Health (NYSDOH).

2.1 Water Column

Water column monitoring will include sampling and analytical testing to evaluate any solids and/or PCB releases; details on each parameter are provided in this section, along with the discussion of the designated water column monitoring locations. The standard operating procedure (SOP) for water column monitoring is provided in Attachment C to the CQAP.

Stratification occurs in the Lower Grasse River when colder water with greater specific conductivity (relative to the Grasse River water) from the St. Lawrence River enters into and moves upstream along the bottom of the Lower Grasse River. The water column monitoring approach will be altered when stratification is present to collect samples that are as representative of the Grasse River water as possible. Therefore, water quality parameters (i.e., temperature and specific conductivity) will be obtained before each monitoring event at 0.2 and 0.8 times the total water column depth from each sampling station in the Grasse

River with depths greater than 5 feet to assess for the presence of stratification. Based on previous evaluations, differences of 3 degrees Celsius in water temperature or 20 microSiemens per centimeter in specific conductivity between the two water masses will be used to identify the existence of stratification (Alcoa, May 2006). If stratification is not present at a given location, water column grab samples will be collected at 0.2 and 0.8 times the total water column depth and composited to form one sample. If stratification is present at a given location, water column grab samples will only be collected above the stratification (i.e., 0.2 times the total water column depth) for laboratory analysis.

2.1.1 Solids

Grab samples will be collected once daily at each of the monitoring locations during near shore debris removal, dredging, floodplain excavation, backfilling and capping and submitted for total suspended solids (TSS) analysis using Method SM2540D. If stratification is not present at a given location, water column grab samples will be collected at 0.2 and 0.8 times the total water column depth and composited to form one sample for laboratory analysis. If stratification is present at a given location, water column grab samples will only be collected above the stratification (i.e., 0.2 times the total water column depth) for laboratory analysis. In areas of shallow water (i.e., upstream portion of the river where maximum water depths are less than 5 feet), samples will be collected at 0.5 times the total water column depth.

2.1.2 PCB (Aroclor)

Grab samples will be collected once daily during near shore debris removal, dredging, floodplain excavation, and backfilling from monitoring stations downstream of the operations and submitted for PCB (Aroclor) analysis using the project-specific modified SW-846-8082A. Grab samples will also be collected once a day during the first month of capping activities from monitoring stations downstream of capping activities and submitted for PCB (Aroclor) analysis, with results then reviewed with USEPA, New York State Department of Environmental Conservation (NYSDEC), and Saint Regis Mohawk Tribe (SRMT) to determine the need for additional monitoring. If stratification is not present at a given location, water column grab samples will be collected at 0.2 and 0.8 times the total water column depth and composited to form one sample for laboratory analysis. If stratification is present at a given location, water column grab samples will only be collected above the

stratification (i.e., 0.2 times the total water column depth) for laboratory analysis. In areas of shallow water (i.e., upstream portion of the river where maximum water depths are less than 5 feet), samples will be collected at 0.5 times the total water column depth.

2.1.3 Metals and PAHs

Grab samples will be collected once a week for the first month of near shore construction activities from the near-field station and submitted for analyses for lead (via SW-846-6020A), mercury (via SW-846-7470A), and polycyclic aromatic hydrocarbons (PAHs, including anthracene, benz(a)anthracene, 2-methylnaphthalene, naphthalene, phenanthrene, and pyrene via SW-846-8270D-SIM). The analytes selected for testing include only those detected during the River and Sediment Investigation (RSI) Phase I (Ecology and Environment, Inc. [E&E], October 1992) that have associated ambient water standards listed in the NYSDEC Technical and Operational Guidance Series (TOGS). If stratification is not present, water column grab samples will be collected at 0.2 and 0.8 times the total water column depth and composited to form one sample for laboratory analysis. If stratification is present, water column grab samples will only be collected above the stratification (i.e., 0.2 times the total water column depth) for laboratory analysis.

2.1.4 Receipt of Results

TSS, PCB (Aroclor), metals, and PAH samples will be submitted to the selected laboratory for analysis, and results will be requested on an accelerated turn-around-time (TAT, 24 hours). Water column sampling will be initiated no sooner than approximately 2 hours after the start of in-water activities on any given day. Note that sample shipping transport times, the time of day when the sample is collected and submitted, and weekend work may impact the selected laboratory's ability to meet this TAT, and sample results may not be available for 48 to 72 hours after collection.

2.1.5 Water Column Monitoring Stations

Water column monitoring stations will be established at three stations at the midpoint across the Grasse River and one location in the St. Lawrence River as listed below (Figure C-D2-1).

- Upstream – Fixed station established upstream of all dredging or capping areas at approximately T0 (this is a shallow water station with less than 5 feet of water present)
- Near-field – Mobile station that will be adjusted as activities advance downstream; station will be established approximately 1,000 feet downstream of collective in-river work activities during each monitoring event (e.g., the station will be established mid-river 1,000 feet downstream of the furthest downstream work activities)
  - This station will be eliminated when construction activities reach T71 as the location will then be replaced by the station in the St. Lawrence River (see below)
- T71 – Station established at the historic water column sampling location WC013 (located just upstream of the Grasse River mouth)
  - This station will be eliminated when construction activities reach T69 (within 1,000 feet of the station) as the location will then be replaced by the near-field mobile station
- Far-field or St. Lawrence River – Station established at the water column sampling location in the St. Lawrence River along the southern shoreline immediately downstream of the Grasse River mouth

Advisory and corrective action levels have been identified as described in Section 2.3. The near-field location would represent water column conditions closest to the remedial activities (i.e., worst-case scenario).

2.2 Water Intakes

Monitoring will be performed at the Alcoa East Plant and SRMT water intakes located downstream of the Grasse River mouth at approximately 0.75 and 4.5 miles, respectively (Figure C-D2-2). A single baseline monitoring event will be performed 2 to 4 weeks before in-water work begins to assess levels prior to remediation for the purpose of evaluating the corrective action level. During near shore debris removal and dredging, floodplain excavation, backfilling, and the first month of capping activities, water intake monitoring will include daily collection of grab samples from the raw (untreated) sampling port inside the Alcoa East Plant and the raw (untreated) and treated water within the SRMT Water

Treatment Building (i.e., a total of three daily samples) for PCB (Aroclor) analysis. Turbidity measurements will also be recorded at the time of sampling at each intake location using a real-time meter. Turbidity measurements will be obtained daily during capping at each intake location. Samples will be submitted to the selected laboratory for PCB (Aroclor) analysis using SW-846-8082A. Based on SRMT's request, samples will also be collected monthly from the SRMT treatment building (untreated and treated water) and submitted for PCB congener analysis using SW-8270D-SIM. These samples will be submitted to the selected laboratory for analysis with results requested on a standard analytical TAT because the data will be used for informational purposes only.

Results for PCB Aroclor samples will be requested on an accelerated TAT (as noted in Section 2.1, sample results may not be available for 48 to 72 hours after collection). Samples will be collected approximately 4 to 6 hours after the start of in-water activities on any given day. The SOP for water intake monitoring is provided in Attachment C to the CQAP.

2.3 Advisory and Corrective Action Levels

Advisory and/or corrective action levels have been established for each monitoring criterion. These levels have been developed through consultation with USEPA, NYSDEC, and SRMT, and in consideration of the Grasse River pilot projects and other New York State projects. A summary of these levels is provided in this section.

2.3.1 Water Column Monitoring

Advisory and/or corrective action (compliance) levels and associated response actions have been identified for the in-river monitoring. These levels and actions are identified based on the water monitoring location.

Near-field:

The water quality criteria at the near-field monitoring station (mobile station) will be considered an advisory level for evaluating potential operational adjustments for near shore debris removal and dredging, floodplain removal, and backfilling and also main channel capping. This station will serve as a diagnostic tool. The near-field monitoring station advisory levels will include the following:

- TSS concentrations of 100 milligrams per liter (mg/L) above the upstream station (i.e., ambient conditions)
- Total PCB concentrations of 0.500 micrograms per liter (µg/L)
- Metals – As required by NYSDEC, less than standard specified in Title 6 of the New York Codes, Rules and Regulations (6 NYCRR) Part 703.5 for Class C (chronic aquatic);[1] assessed by comparing the sample result at the far-field station against the levels for the following:
  - Lead (dissolved) – 2.3 µg/L
    - Lead Formula = {1.46203 – [ln (hardness) (0.145712)]} exp (1.273 [ln (hardness)] – 4.297)
    - Hardness determined as an average of the means from Reaches 4 through 8 in the RSI Phase I = 63 mg/L
  - Mercury (dissolved) – 0.77 µg/L
- PAHs – Less than the standard specified in TOGS 1.1.1 for Class C (acute aquatic);[2] assessed by comparing the sample result at the far-field station against the following levels:
  - Anthracene – 35 µg/L
  - Benz(a)anthracene – 0.23 µg/L
  - 2-Methylnaphthalene – 42 µg/L
  - Naphthalene – 110 µg/L
  - Phenanthrene – 45 µg/L
  - Pyrene – 42 µg/L

Exceedance of the advisory levels will trigger work review/evaluation and response actions, as appropriate. An evaluation will be conducted to assess the cause of the exceedance and the need for operational changes or other response actions. Such an evaluation may result in one or more of the following actions:

[1]Available at: https://govt.westlaw.com/nycrr/Document/I4ed90418cd1711dda432a117e6e0f345?viewType=FullText&originationContext=documenttoc&transitionType=CategoryPageItem&contextData=(sc.Default)

[2] Available at: http://www.dec.ny.gov/docs/water_pdf/togs1112.pdf

- Additional monitoring as necessary to investigate the cause of the exceedance such as collecting and analyzing individual grab samples from 0.2 and 0.8 times the total water column depth
- A review of work operations and best management practices (BMPs), including investigation of the cause of the exceedance
- Identification of possible operational adjustments or BMPs (to the extent practicable) to reduce the concentrations in the water column

Following such evaluation, Arconic will discuss with USEPA, NYSDEC, and SRMT whether operational changes or other response actions are warranted to address the exceedance of the advisory level. This may include active field refinements in operations or equipment while continuing operations. However, exceedance of the advisory levels at the near-field station will not result in a slowdown or shutdown of operations. After one month of operations, Arconic will review the monitoring data with USEPA, NYSDEC, and SRMT, and may request moving the near-field monitoring location farther downstream or adjusting the monitoring frequency. Any additional proposed changes to the construction activities will be documented for approval as described in the CQAP.

T71:

At the T71 monitoring station, exceedance of 0.500 ug/L total PCBs advisory level will trigger work review/evaluation and response actions, as appropriate. An evaluation will be conducted to assess the cause of the exceedance and the need for operational changes or other response actions. Such an evaluation may result in one or more of the following actions:

- Additional monitoring as necessary to investigate the cause of the exceedance such as collecting and analyzing individual grab samples from 0.2 and 0.8 times the total water column depth
- A review of work operations and BMPs, including investigation of the cause of the exceedance
- Identification of possible operational adjustments or BMPs (to the extent practicable) to reduce the concentrations in the water column

Following such evaluation, Arconic will discuss with USEPA, NYSDEC, and SRMT whether operational changes or other response actions are warranted to address the exceedance of the advisory level. This may include active field refinements in operations or equipment while continuing operations; however, exceedance of the total PCBs advisory level at the T71 monitoring station will not result in shutdown of operations in this situation.

SLR:

The SLR monitoring station will be used as the compliance location. Exceedance of 0.500 ug/L total PCBs may trigger stop work and examining the BMPs employed and making appropriate modifications.

2.3.2 Water Intake Monitoring

Corrective action (compliance) levels and associated response actions have been identified for the Alcoa East Plant and SRMT intakes.

- Turbidity – 100 nephelometric turbidity units greater than the baseline level
  - Assessed by comparing the reading during remediation against the baseline reading
- PCB (Aroclor) – 0.500 µg/L
  - Assessed by comparing the sample result against the corrective action level

Exceedances of any of these criteria will initially result in a review of the next-day sample results to determine whether the exceedance still exists. If the second sample verifies the exceedance, a review of the remediation activities will be immediately conducted, with adjustments made as needed in accordance with the Contingency Plan provided in Appendix L of the FDR.

If initial monitoring results indicate no exceedances of the corrective action triggers listed in this section, Arconic may work with USEPA, NYSDEC, and SRMT to modify the frequency or duration of monitoring at certain locations. Any additional proposed changes to the construction activities will be documented for approval as described in the CQAP.

3 AIR MONITORING

Air monitoring will be conducted during all intrusive activities including near shore debris removal and dredging, floodplain excavation, backfilling, contaminated sediment dewatering and processing, and landfill operations. In addition, air monitoring will be performed during material handling activities associated with backfill or capping activities around the staging area. Therefore, air monitoring will be initiated at the start of ground intrusive activities and will continue through the in-water near shore dredging and debris removal activities and completion of capping. Note that no work is planned to be performed within 20 feet of potentially exposed individuals or structures; Arconic will consult with USEPA, NYSDEC, NYSDOH, and SRMT if it becomes necessary to perform work within these limits. Air monitoring will include real-time continuous particulate and VOC measurements and continuous air sampling for PCB analysis as described in the following sections. The SOP for air monitoring and typical equipment specification sheets are provided in Attachment C to the CQAP.

3.1 Real-Time Meter Monitoring

Real-time meter monitoring will include daily continuous particulate (less than 10 microns [PM_{10}]) and VOC monitoring. Monitoring will be performed in the vicinity of sources of potential particulate emissions during intrusive activities including grading, sediment and soil handling and processing at the staging area and disposal at the secure landfill (SLF). Particulate monitoring will be performed during backfill and cap material handling at the staging area. Note that monitoring is not included for the near shore dredge and backfilling because removal and placement of these materials will be performed in-the-wet within small work areas, thus reducing the potential for particulate releases.

Monitoring will be conducted using real-time meters to obtain continuous readings for determining the 24-hour average. The meters selected for use during these monitoring activities will be capable of calculating 15-minute running average concentrations. Meters will be checked periodically during the day by on-site personnel. The meters will be equipped with audible alarms that will indicate whether concentrations exceed an established level. Data from these meters will be transmitted to the on-site construction management team using telemetry. In addition to the alarms, the telemetry system will

notify on-site personnel of exceedances of established levels via text message or email. This will allow any potential exceedances to be investigated immediately. If visible dust is observed leaving the work area, the appropriate meters will be checked as soon as possible after the dust has been observed.

3.2 Polychlorinated Biphenyls

PCB monitoring will be performed in the vicinity of potential sources of PCB emissions during near shore debris removal and dredging, floodplain excavation, sediment handling and processing at the staging area, and disposal at the SLF. PCB monitoring will be performed using continuous 24-hour air samplers. High-volume sampling will be performed at fixed locations around the staging area and SLF with available power, and low-volume samplers will be used for the mobile locations to be established in the vicinity of the near shore dredging and floodplain removal areas (referred to as the dredge corridor) or at fixed locations at the staging area where no power source is available.

High-volume sampling will be conducted in accordance with USEPA Method TO-4A. PCB samples will be collected using high-volume air samplers fitted with quartz fiber filters and sorbent cartridges. The high-volume polyurethane foam (PUF) sampler located at each monitoring station will be operated at a flow rate of 200 to 300 liters per minute (L/min), with a sampling period of approximately 24 hours. High-volume samplers require a continuous power source; therefore, these locations will be stationary throughout the required monitoring period.

Low-volume sampling will be conducted in accordance with USEPA Method TO-10A. PCB samples will be collected using a low-volume personal sampling pump equipped with a glass cylinder containing a polyurethane sorbent (i.e., PUF plug). Samples will be collected at a flow rate of approximately 5 L/min, with a sampling period of approximately 24 hours. Low-volume samplers are battery powered and can therefore be relocated.

Air samples will be submitted for PCB (Aroclor) analysis using SW-846-8082. Results will be requested on an accelerated TAT. A TAT of 48 hours is the fastest time possible considering the analytical testing requirements; however, sample shipping transport times, the time of

day when the sample is collected and submitted, and weekend work may impact the selected laboratory's ability to meet this TAT, and sample results may not be available for 72 to 96 hours after collection.

3.3 Air Monitoring Locations

Air monitoring stations will be established at the SLF, staging area, and in proximity to the near shore dredge and floodplain removal areas. Historical determination of the prevailing wind direction has been made based on local Massena/Federal Aviation Administration Airport Meteorological Station No. 94725. The station at the airport is located less than 1.5 miles south of the staging area adjacent to the Route 131 Bridge. Using data from this station, a wind rose was developed using wind direction data averaged from 2010 to 2014 for the months of April to October. The historical predominant wind direction was determined to be from the southwest. The upwind and downwind locations will be determined daily using data from the airport based on the dominant wind through observation of meteorological conditions and data (e.g., wind speed and direction, wind gust speed and direction, temperature, and humidity).

3.3.1 SLF Air Monitoring Stations

A total of four fixed air monitoring stations will be established around the SLF based on potential receptor locations, historical predominant wind direction, and power availability. In addition, two portable station setups will be established in areas where there is no power available. Figure C-D3-1 provides the approximate location of the SLF air monitoring stations (including two locations for portable station setup). The upwind and downwind locations will be determined daily based on the meteorological data and will be used to assess corrective action levels (Section 3.4).

At the direction of NYSDEC and NYSDOH, air monitoring will be performed 24 hours per day, 7 days per week at the fixed monitoring stations. The portable station setups will only be used during days of active construction at the landfill. As requested by NYSDEC and NYSDOH, the portable monitoring stations will be turned on and off at the beginning and end of each active work period, respectively.

The wind direction will be used to determine whether PCB samples from the portable meters will be analyzed. The wind direction will be reviewed via electronic means and field verified periodically as possible (target approximately every 2 hours) throughout working hours each day through a windsock near the SLF. If, at any point during the run time of the portable stations the wind direction indicates these stations to be in a downwind direction (see Table C-D3-1) or the wind is calm that particular day (i.e., meteorological data from the air reports conditions are calm and there is no wind speed or gusts), PCB samples from either or both portable stations will be analyzed. The four fixed locations will be analyzed for PCBs daily.

Table C-D3-1, below, summarizes the downwind station in consideration of wind direction at 22.5-degree intervals and indicates where analysis of PCBs at a portable monitor would be invoked.

Table C-D3-1
SLF – Summary of Wind Direction and Associated Downwind Stations

Wind Direction	Downwind Fixed Station				Portable Station
	SLF-Air 1	SLF-Air 2	SLF-Air 3	SLF-Air 4	
N	X				
NNE	X				
NE	X				
ENE					X (A)
E					X (B)
ESE					X (B)
SE					X (B)
SSE				X	X (B)
S				X	X (B)
SSW				X	
SW			X		
WSW			X		
W			X		
WNW			X		
NW		X			
NNW		X			

Note:
SLF: secure landfill

PM_{10} and VOC data from all stations (including portable stations) will be reported daily.

3.3.2 Staging Area Air Monitoring Stations

Air monitoring stations will be established upwind and downwind of the staging area sediment removal and processing areas. Figure C-D3-1 provides the approximate location of the staging area air monitoring stations (four stations). The upwind and downwind locations will be determined daily based on the meteorological data and will be used to assess corrective action levels (Section 3.4).

Table C-D3-2, below, summarizes the downwind station in consideration of wind direction at 22.5-degree intervals.

Table C-D3-2
Staging Area – Summary of Wind Direction and Associated Downwind Stations

Wind Direction	Downwind Station			
	SA-Air 1	SA-Air 2	SA-Air 3	SA-Air 4
N			X	
NNE		X		
NE		X		
ENE		X		
E	X			
ESE	X			
SE	X			
SSE		X		
S				X
SSW				X
SW				X
WSW				X
W			X	
WNW			X	
NW			X	
NNW			X	

Note:
SA: staging area

Air monitoring associated with the staging area will be performed continuously during the first month of activities (i.e., 24 hours per day, 7 days per week including construction and non-construction times). This continuous monitoring will result in data collection during both construction and non-construction periods; based on these data, Arconic will then either submit a request to the Agencies to modify monitoring to the period of construction only (i.e., only during days of active operation) or will maintain the continuous monitoring efforts. PCBs samples from all locations will be sent for analysis each monitoring day. PM_{10} and VOC data from all stations will be reported each monitoring day.

3.3.3 Near Shore Dredging and Floodplain Removal Mobile Air Monitoring Stations

Low-volume PCB sampling locations associated with the near shore dredging and floodplain removal and located in the "dredge corridor" (e.g., along the river shoreline) will be determined in the field based on potential receptors and their proximity to the dredging and floodplain removal areas. Up to three monitoring station will be setup along the shoreline between the nearest receptor and dredging/removal activities. Prevailing wind direction is from the southwest; therefore, monitoring stations will be established considering this predominant wind direction. Arconic developed a memorandum that established guidelines and included a series of figures to identify the target mobile air station locations by target dredge area or transect. This document was reviewed/approved by USEPA, NYSDEC, NYSDOH, and SRMT, and is included in Appendix B. The location of the station will be recorded, so the distance from the dredging/removal activity can be determined. The monitoring locations will be moved to new areas as operations proceed downstream in accordance with Appendix B. These target locations may need to be adjusted based on field conditions, access, etc.

Air monitoring associated with the near shore dredging/floodplain removal will be performed only during days of active operation. PCB samples will be sent for analysis each day of operation.

3.4 Corrective Action Levels

Corrective action levels have been established for each monitoring criterion. These levels have been developed in consideration of the NYSDOH Generic Community Air Monitoring Plan provided in DER-10 (NYSDEC, May 2010) and Grasse River pilot projects. The corrective action levels will apply at the downwind location only; in instances of calm, the corrective action levels would be considered at all locations. The overall predominant wind direction will be determined daily based on the meteorological data; however, in the event of any exceedances of the of the particulate and VOC criteria noted below, the predominant wind direction will be determined at the time of the exceedance to identify the upwind and downwind directions. The following summarizes the corrective action levels:

- Particulate (PM_{10}) – 0.150 milligrams per cubic meter (mg/m^3) above the upwind location level for a 15-minute period:
  - If the downwind PM_{10} level is 0.100 mg/m^3 above the upwind location level for the 15-minute period or if airborne dust is observed leaving the work area, then dust-suppression techniques must be employed. Work may continue with dust-suppression techniques provided that downwind PM_{10} particulate levels do not exceed 0.150 mg/m^3 above the upwind level and no visible dust is migrating from the work area.
  - If after implementation of dust-suppression techniques the downwind PM_{10} levels are more than 0.150 mg/m^3 above the upwind location level, work must be stopped and site activities must be evaluated. Work may resume only if dust-suppression measures and other controls are successful in reducing PM_{10} levels to less than 0.150 mg/m^3 above background and if no visible dust is observed leaving the site.
  - An initial level of 0.100 mg/m^3 (15-minute average) at any of the monitoring stations will be established as a conservative assessment level. Readings greater than this conservative assessment level will result in on-site personnel performing a review of the background (upwind perimeter) site level. If the downwind level is determined to be more than 0.100 mg/m^3 above background (upwind perimeter), dust-suppression techniques will be employed to avoid an exceedance of the corrective action level.
- VOC – 25 parts per million (ppm) at the downwind station for a 15-minute period
  - If the downwind VOC level is 5 ppm above the upwind station for the 15-minute period, then work activities must be investigated and monitoring continued. If the total organic vapor level readily decreases (per instantaneous readings) below 5 ppm more than background, work activities can proceed with continued monitoring.
  - If the downwind VOC levels persist at 5 ppm over the upwind station level but are less than 25 ppm, work activities must be halted, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities can resume provided that the VOC level 200 feet downwind of the exclusion zone or half the distance to the nearest potential

receptor or residential/commercial structure, whichever is less (but in no case less than 20 feet), is below 5 ppm over background for the 15-minute average.
  - If VOC levels at any station are above 25 ppm, activities must be shut down.

- PCB (Aroclor) – 0.100 microgram per cubic meter

Air analytical results will be received on an accelerated schedule from the selected laboratory. Exceedances of any of these criteria will result in an immediate review of construction activities, with adjustments made as needed in consultation with the remedial contractor and in accordance with the Contingency Plan provided in Appendix L of the FDR. For the real-time meters, the first step of this review will be to evaluate the result to assess whether it is site related or an issue with the meter (e.g., high humidity impacting the meter and readings) or local conditions (e.g., mowing in the vicinity of the meter). Once an exceedance has been verified to be site related, the Construction Manager will immediately be notified and an investigation will be performed to identify the conditions causing the exceedance. The Construction Manager will evaluate these factors and identify the likely cause of the exceedance in order to appropriately respond to the issue. An email will be issued to the Agencies by the next business day documenting the exceedance and the results of the evaluation.

If initial monitoring results indicate no exceedances of the corrective action triggers listed in this section, Arconic may work with USEPA, NYSDEC, and SRMT to modify the frequency or duration of monitoring at certain locations. Any additional proposed changes to the construction activities will be documented for approval as described in the CQAP.

4 DATA QUALITY ASSURANCE

Data QA for sampling will be conducted consistent with the Quality Assurance Project Plan (QAPP) Worksheets provided in Attachment C to the CQAP as well as the worksheets in the Pre-Design Investigation (PDI) QAPP (Alcoa, October 2013).

4.1 Water

The bullets below list the applicable worksheets along with a reference to the specific document.

- Worksheet No. 12: QA/QC Measurement Performance Criteria (Attachment C to the CQAP)
- Worksheet No. 15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits (Attachment C to the CQAP)
- Worksheets No. 19 and 30: Sample Containers, Preservation, and Hold Times (Attachment C to the CQAP)
- Worksheet No. 20: Field QC Summary (Attachment C to the CQAP)
- Worksheet No. 22: Field Equipment Calibration, Maintenance, Testing, and Inspection (Attachment C to the CQAP)
- Worksheet No. 23: Analytical SOPs (Attachment C to the CQAP)
- Worksheet No. 24: Analytical Instrument Calibration (Attachment C to the CQAP)
- Worksheet No. 25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection (Attachment C to the CQAP)
- Worksheets No. 26 and 27: Sample Handling, Custody, and Disposal (Attachment C to the CQAP)
- Worksheet No. 28: Analytical Quality Control and Corrective Action (Attachment C to the CQAP)
- Worksheet No. 29: Project Documents and Records (Alcoa, October 2013)
- Worksheets No. 31, 32, and 33: Assessments and Corrective Action (Alcoa, October 2013)
- Worksheet No. 34: Data Verification and Validation Inputs (Alcoa, October 2013)
- Worksheet No. 35: Data Verification Procedures (Alcoa, October 2013)
- Worksheet No. 36: Data Validation Procedures (Alcoa, October 2013)
- Worksheet No. 37: Data Usability Assessment (Alcoa, October 2013)

QA/QC samples specific to the water monitoring outlined in the EMP are as follows:

- One duplicate sample for PCB (Aroclor), PCB congener, and TSS per 20 samples collected
- One matrix spike/matrix spike duplicate for PCB (Aroclor) and PCB congener per 20 samples collected
- One equipment rinse blank sample at the start of monitoring, one at the end of each day's monitoring activities, and one at the conclusion of monitoring for PCB (Aroclor)

4.2 Air

The bullets below list the applicable worksheets along with a reference to the specific document.

- Worksheet No. 12: QA/QC Measurement Performance Criteria (Attachment C to the CQAP)
- Worksheet No. 15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits (Attachment C to the CQAP)
- Worksheets No. 19 and 30: Sample Containers, Preservation, and Hold Times (Attachment C to the CQAP)
- Worksheet No. 20: Field QC summary (Attachment C to the CQAP)
- Worksheet No. 22: Field Equipment Calibration, Maintenance, Testing, and Inspection (Attachment C to the CQAP)
- Worksheet No. 23: Analytical SOPs (Attachment C to the CQAP)
- Worksheet No. 24: Analytical Instrument Calibration (Attachment C to the CQAP)
- Worksheet No. 25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection (Attachment C to the CQAP)
- Worksheets No. 26 and 27: Sample Handling, Custody, and Disposal (Attachment C to the CQAP)
- Worksheet No. 28: Analytical Quality Control and Corrective Action (Attachment C to the CQAP)
- Worksheet No. 29: Project Documents and Records (Alcoa, October 2013)
- Worksheets No. 31, 32, and 33: Assessments and Corrective Action (Alcoa, October 2013)

- Worksheet No. 34: Data Verification and Validation Inputs (Alcoa, October 2013)
- Worksheet No. 35: Data Verification Procedures (Alcoa, October 2013)
- Worksheet No. 36: Data Validation Procedures (Alcoa, October 2013)
- Worksheet No. 37: Data Usability Assessment (Alcoa, October 2013)

QA/QC samples specific to the air monitoring outlined in the EMP are as follows:

- One field duplicate for PCB (Aroclor) per 20 samples collected

5 REFERENCES

Alcoa (Alcoa, Inc.), April 2002. *Documentation Report – Grasse River Capping Pilot Study*.

Alcoa, May 2006. *Draft Remedial Options Pilot Study Documentation Report*.

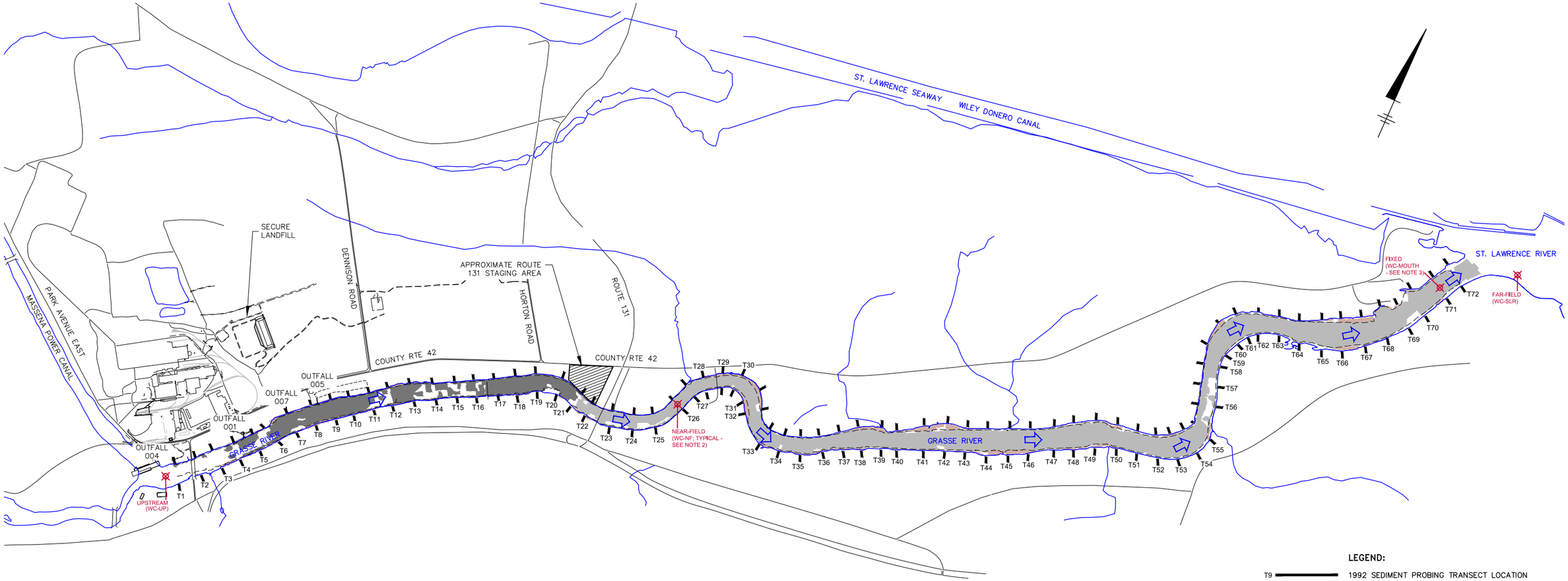
Alcoa, October 2013. *Pre-Design Investigation Quality Assurance Project Plan*.

E&E (Ecology and Environment, Inc.), October 1992. *Phase I – Grasse River: River and Sediment Investigation (Volumes I and II)*. Prepared for Alcoa, Massena, New York.

NYSDEC (New York State Department of Environmental Conservation), May 2010. *DER-10 Technical Guidance for Site Investigation and Remediation*.

USEPA (U.S. Environmental Protection Agency), April 2013. *Record of Decision*. Grasse River Superfund Site, Massena, St. Lawrence County, New York.

FIGURES



LEGEND:

- T9 — 1992 SEDIMENT PROBING TRANSECT LOCATION
- RIVER BOUNDARY
- NEAR SHORE AREA
- DIRECTION OF RIVER FLOW
- MAIN CHANNEL ARMORED CAP
- MAIN CHANNEL SAND CAP
- NEAR SHORE DREDGE/BACKFILL
- UPSTREAM (WC-UP) — APPROXIMATE WATER COLUMN MONITORING STATION

0 1200' 2400'
GRAPHIC SCALE

NOTES:

1. BASE MAP TAKEN FROM PLANIMETRIC MAPPING PREPARED BY LOCKWOOD MAPPING, INC. USING 11/9/92 AERIAL PHOTOGRAPHY. RIVER BOUNDARY PROVIDED BY ANCHOR QEA ON SEPTEMBER 15, 2016, AND SHOULD BE CONSIDERED APPROXIMATE ONLY.
2. WC–NF IS A MOBILE STATION THAT WILL BE ESTABLISHED MID–RIVER APPROXIMATELY 1,000 FEET DOWNSTREAM OF COLLECTIVE IN–RIVER WORK ACTIVITIES DURING EACH MONITORING EVENT. THE EXACT LOCATION WILL BE DETERMINED AT THE TIME OF MONITORING. THIS STATION WILL BE ELIMINATED WHEN CONSTRUCTION ACTIVITIES REACH T71 AS IT WILL BE REPLACED BY WC–SLR.
3. WC–MOUTH IS A FIXED STATION THAT WILL BE ELIMINATED WHEN CONSTRUCTION ACTIVITIES REACH T69 (WITHIN 1,000 FEET OF THE STATION) AS IT WILL BE REPLACED BY WC–NF.

GRASSE RIVER STUDY AREA
MASSENA, NEW YORK
ENVIRONMENTAL MONITORING PLAN

WATER COLUMN MONITORING LOCATIONS

ARCONIC

FIGURE
C-D2-1

CITY: SYRACUSE, NY DIV/GROUP: IMDV/CAD DB: L.POSENAUER PIC/PM: H. VANDEWALKER TM: S. HILL LYR: ON=*; OFF=*REF*
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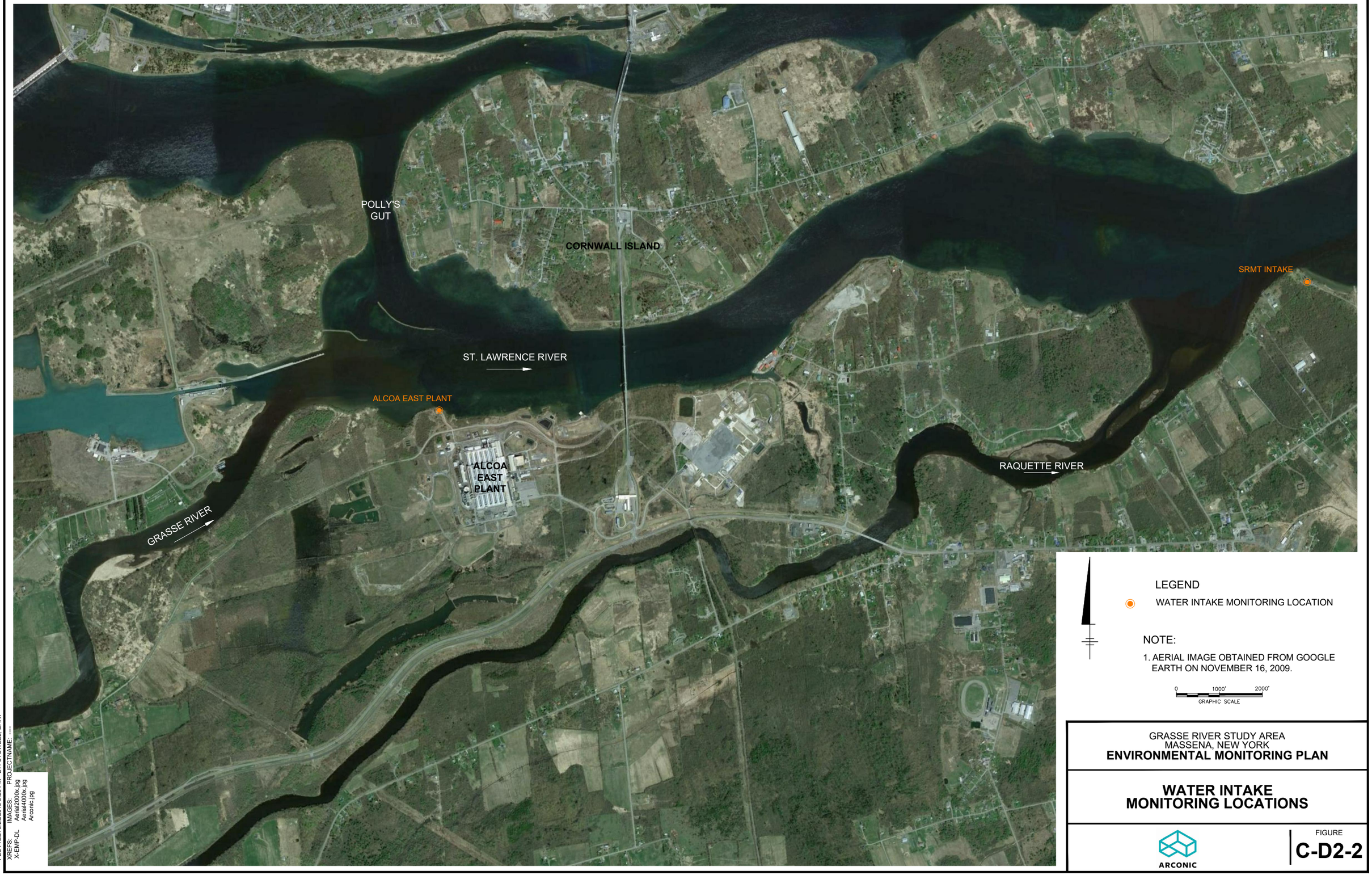

LEGEND

WATER INTAKE MONITORING LOCATION

NOTE:

1. AERIAL IMAGE OBTAINED FROM GOOGLE EARTH ON NOVEMBER 16, 2009.

0 1000' 2000'
GRAPHIC SCALE

GRASSE RIVER STUDY AREA
MASSENA, NEW YORK
ENVIRONMENTAL MONITORING PLAN

WATER INTAKE MONITORING LOCATIONS

ARCONIC

FIGURE
C-D2-2

CITY: SYRACUSE DIV/GROUP: ENVCAD DB: L. FORAKER T. RITSCHEL L. FORAKER LD: PIC: H. VANDEWALKER PM: H. VANDEWALKER TM: S. HILL LYR: ON=*;OFF=*REF*
C:\BIMOneDrive - ARCADIS\BIM 360 Docs\ARCONIC INC\GRASSE RIVER FDR & RFP SUPPORT\2019\B0010886.0036\01-DWG\FIG-C-D2-3-MON-LOCS.DWG LAYOUT: C-D2-2 SAVED: 3/28/2019 3:25 PM ACADVER: 23.0S (LMS TECH) PAGESETUP: C-LD2B-PDF-GMS PLOTSTYLETABLE: PLTFULL.CTB
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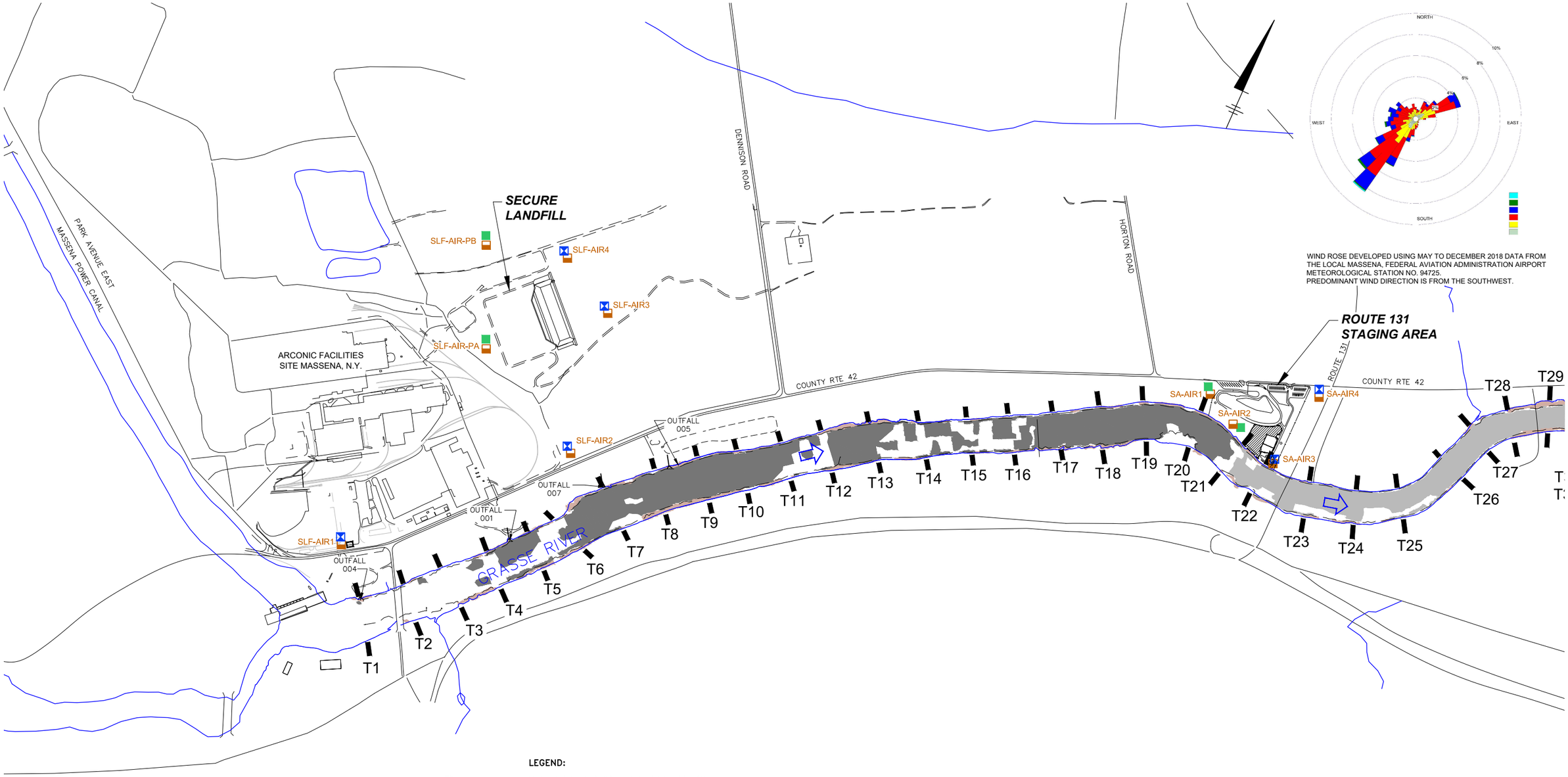
Aerial4000x.jpg
Arconic.jpg



LEGEND:

- T9 — 1992 SEDIMENT PROBING TRANSECT LOCATION
- RIVER BOUNDARY
- NEAR SHORE AREA
- DIRECTION OF RIVER FLOW
- MAIN CHANNEL ARMORED CAP
- MAIN CHANNEL SAND CAP
- NEAR SHORE DREDGE/BACKFILL

APPOXIMATE AIR MONITORING LOCATIONS:

- PARTICULATE AND VOC
- HIGH VOLUME PCB
- LOW VOLUME PCB

NOTES:

1. BASE MAP TAKEN FROM PLANIMETRIC MAPPING PREPARED BY LOCKWOOD MAPPING, INC. USING 11/9/92 AERIAL PHOTOGRAPHY. RIVER BOUNDARY PROVIDED BY ANCHOR QEA ON SEPTEMBER 15, 2016, AND SHOULD BE CONSIDERED APPROXIMATE ONLY.
2. THE FINAL FIXED AIR MONITORING STATION LOCATIONS WILL BE SELECTED IN THE FIELD BASED ON SITE CONDITIONS AND POWER AVAILABILITY.
3. AIR MONITORING STATIONS WILL BE LOCATED IN THE DREDGE CORRIDOR IN CONSIDERATION OF EACH DREDGE MANAGEMENT UNIT (DMU) AND/OR FLOODPLAIN REMOVAL AREA. UP TO 3 MONITORING STATIONS WILL BE ESTABLISHED FOR EACH DMU OR GROUP OF ADJACENT DMUs. THESE MOBILE MONITORING STATIONS WILL BE MOVED AS DREDGING/REMOVAL PROCEEDS DOWNSTREAM. THESE STATIONS ARE NOT SHOWN ON THIS FIGURE. THERE ARE ALSO 4 STATIONS UPSTREAM OF T7 THAT WILL BE MONITORED DURING REMOVAL AT T1N THAT WILL SUPPLEMENT THE DREDGE CORRIDOR STATIONS; THESE STATIONS ARE NOT SHOWN ON THIS FIGURE. ADDITIONAL DETAILS ARE PROVIDED IN APPENDIX B.

GRASSE RIVER STUDY AREA
MASSENA, NEW YORK
ENVIRONMENTAL MONITORING PLAN

AIR MONITORING LOCATIONS

ARCONIC

FIGURE
C-D3-1

CITY: SYRACUSE, NY DIV/GROUP: IMDV/CAD DB: L.POSENAUER PIC/PM: H. VANDEWALKER TM: S. HILL LYR: ON=*; OFF=*REF*
C:\BIM\OneDrive - ARCADIS\BIM 360 Docs\ARCONIC INC\GRASSE RIVER FDR & RFP SUPPORT\2019\B0010886.0036\01-DWG\FIGs-C-D2-1 AND 2-MON-LOCS.dwg LAYOUT: C-D3-1 SAVED: 3/28/2019 2:50 PM ACADVER: 23.0S (LMS TECH) PAGESETUP: C-LD2B-PDF-GMS PLOTSTYLETABLE:
PLTFULL.CTB PLOTTED: 3/28/2019 3:28 PM BY: STOWELL, GARY
XREFS:
X-BASE2
X-EMP-DL

APPENDIX A
JANUARY 2009 ICE MONITORING SCOPE

Proposed River Ice Monitoring Scope for 2008-2009

Component	Current Scope	Proposed Change(s)	Rationale for Change(s)
1. Climatological and River Stage Data Compilation and Review	• Compile air temperature, precipitation, river stage and river flow data • Periodically through winter, daily during breakup	• Add daily staff gage readings at Main Street Bridge, during freeze-up and breakup only	• Provides additional source of flow estimate during ice transition periods
2. River Observation/Documentation	• Observe/document river ice conditions over the winter, with focus on freeze-up and breakup • 15 locations (7 lower / 8 upper) • Monthly from December to time of breakup, daily during breakup	• Reduce to 5 locations (Haverstock Road, , Rte 131 Bridge, Capping Pilot Study Area, Alcoa Bridge, Rte 37 Bridge) • Reduce frequency to once during freeze-up; continue daily during breakup	• Conditions don't change much during winter • Selected locations will provide coverage needed to characterize ice breakup
3. Ice Thickness Measurements / Predictive Modeling	• Monitor ice thickness during the winter • Simulate growth and decay of ice cover using a model • 5 locations (3 lower / 2 upper) • Twice per season (mid-winter and prior to breakup)	• Reduce frequency to once per season (prior to breakup) • Reduce to 3 locations (Rte 131 Bridge, Outfall 001 area, Rte 37 Bridge)	• Historical correlation of measurements to simulation results has been acceptable • Mid-winter measurements not necessary • Selected locations will provide data needed to understand ice cover conditions prior to breakup
4. TSS Monitoring During Ice Breakup	• Quantify solids loading to the lower river during ice breakup • 2 locations (Main St. Bridge, Alcoa Bridge) • Minimum of two times per day during breakup	• Eliminate from program	• Data shows increased solids concentrations during ice breakup relative to non-event conditions, but higher solids levels are maintained for only a short duration (i.e., a few days) and, thus, do not significantly affect total solids loading to the lower river. • Additional measurements will not significantly alter rating curve developed for the site.

APPENDIX B
DREDGE CORRIDOR AIR MONITORING STATION GUIDELINES AND TARGET LOCATIONS

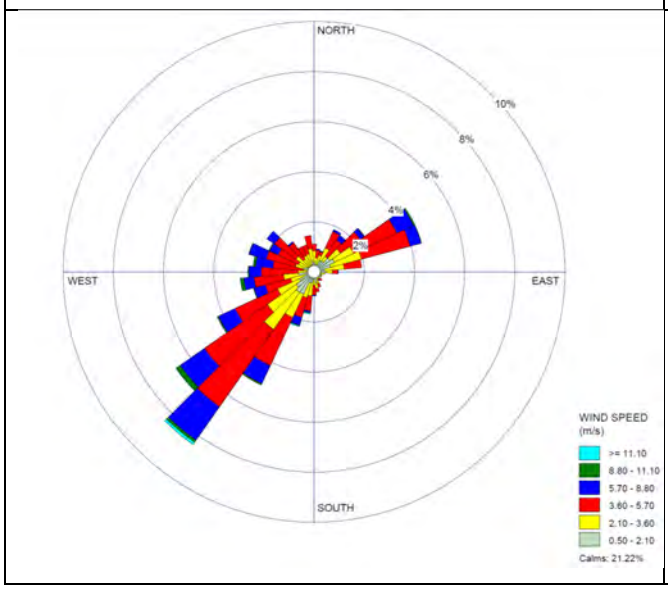
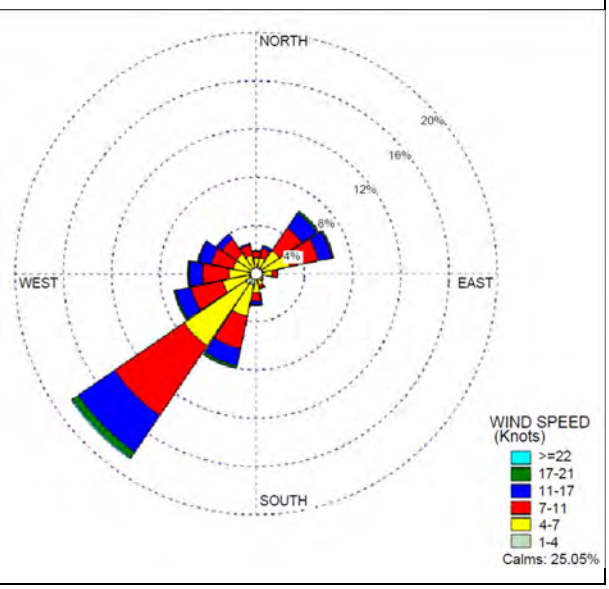
Grasse River Remediation: Dredge Corridor Air Monitoring Station Guidelines and Target Locations

As described in the Grasse River Final Design Report (FDR), mobile air monitoring stations will be established surrounding the near shore dredge and floodplain removal areas along the river shoreline (i.e., "dredge corridor"). This memorandum describes the guidelines used to determine where these stations will be located in the dredge corridor in consideration of each of the dredge management units (DMUs) and/or floodplain removal areas. Up to three monitoring stations will be set up along the shoreline between the nearest receptors and dredging/removal activities for each DMU or group of adjacent DMUs. These mobile monitoring stations will be moved as dredging/removal proceeds downstream. Additional details on the dredge corridor air monitoring program are provided in the FDR (see specifically the Community Health and Safety Plan [CHASP] and Environmental Monitoring Plan [EMP]). An evaluation will be performed on the data received throughout implementation, and based on the results, Arconic may work with the Agencies to modify the program (e.g., frequency, locations, etc.).

The following provides the guidelines used to select the air monitoring station locations, in order of priority. These guidelines were developed in consideration of site-specific monitoring requirements as well as the selection criteria used for the Hudson River remediation project (Anchor QEA, May 2012). These guidelines will be used by field personnel to cite actual monitoring locations as shown on the attached figures; however, adjustments may be needed at the time of equipment deployment based on forecasted weather conditions, actual site conditions, property access, etc. as noted below.

- <u>Predominant historic wind direction and receptor locations</u>: The primary guideline considers the predominant historic wind direction and receptor locations (i.e., off-site receptors such as locations where residences, businesses, and off-site workers not directly involved with remedial activities may be present for a reasonable period of time).

  Historic wind data as well as data from last year's construction activities (2018) were evaluated to develop wind roses to determine the predominant wind direction. The wind roses were developed using data from the Massena, Federal Aviation Administration Airport Meteorological Station No. 94725 (station located less than 1.5 mile south of the staging area). The wind roses using 2018 and historical (2010 to 2014) data are provided below.

May to December 2018 Data	April to October 2010 to 2014
	

Based on this information, as well as site observations, the predominant historic wind direction is from the southwest. The secondary wind directions include from the northeast and northwest. Calm conditions accounted for 20-25% of the data, and wind coming from the southeast was rare. The likely receptor locations were identified using aerial imagery and site-specific knowledge. The target air monitoring station locations were then selected based by evaluating the receptor locations in combination with the predominant historic wind directions. Placement of these portable stations in the field at the time of deployment will also consider forecasted wind directions as possible.

- Surrounding field conditions and environment: The secondary guideline is the United States Environmental Protection Agency (USEPA) and United States Army Corps of Engineers (USACE) criteria regarding ambient particulate sampling systems (Anchor QEA, May 2012). These criteria include the following for positioning the sampler: height of sampler inlet above ground (2 to 15 meters); distance of sampler from trees (greater than 20 meters); distance from sampler to obstacle at least twice the height of the obstacle above the sampler; unrestricted airflow to sampler; roof placement greater than 2 meters from any wall and no nearby flues that may impact sampling; sufficient separation of the sample inlet from roads; and avoidance of locating particulate samplers in unpaved areas unless vegetative cover is present to mitigate impacts of locally dispersed dust. Each of these criteria was considered in selecting the target air monitoring station locations. It may not be possible to comply with all criteria in some areas given topography and land use (e.g., completely wooded area adjacent to a DMU).
- Location of other air monitoring equipment: The third guideline includes consideration of the overall site air monitoring equipment locations and the proximity of the receptors to be protected in the dredge corridor. A number of air monitoring stations will be established for the Route 131 Staging Area (4 stations), Arconic's Secure Landfill (6 stations), and remedial efforts at T1 north (4 additional stations). These stations were reviewed to assess their use to protect adjacent receptors from any potential impacts from the dredge corridor operations.
- Logistical considerations: The fourth guideline is logistical considerations. These considerations include site or area accessibility, safety, security of the equipment, and property ownership and access agreements. All of these considerations will be evaluated in locating the air monitoring stations. Arconic will work to obtain access from private property owners as needed; if access is not granted, the station will either re-located or not established if a suitable alternate area cannot be identified.

These four guidelines were considered in locating the mobile air stations for each of the removal areas and transects. The attached figures illustrate the proposed target locations for the air monitoring stations based on removal areas and/or by river transect (T1 through T72). The station location clusters shown for each DMU or group of adjacent DMUs are independent of shoreline and would be established in each area whether work is being performed on the north shore and/or south shore as applicable. The air monitoring crew will closely coordinate with the contractor/engineering oversight during operations to establish the stations at the necessary locations as work progresses downstream.

Reference:

Anchor QEA, LLC, May 2012. Hudson River PCBs Superfund Site – Phase 2 Remedial Action Monitoring Quality Assurance Project Plan, prepared for General Electric in conjunction with Environmental Standards, Inc.

FIGURE 1

Grasse River Arconic Portal

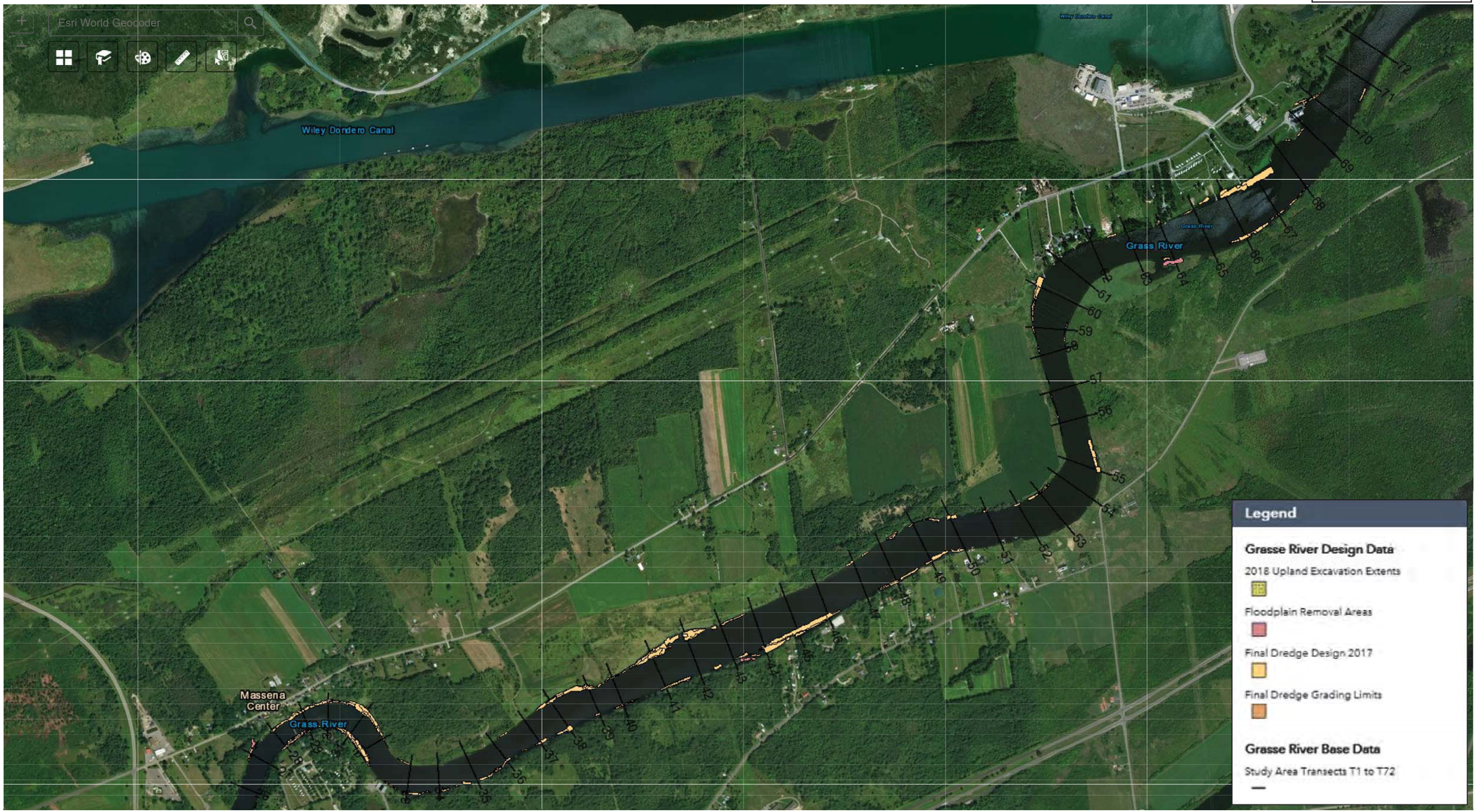
GENERAL VIEW SHOWING DMUs, FP REMOVAL AREAS, AND RECEPTORS

0.2mi
-74.780 44.964 Degrees

N

FIGURE 2

Grasse River Arconic Portal



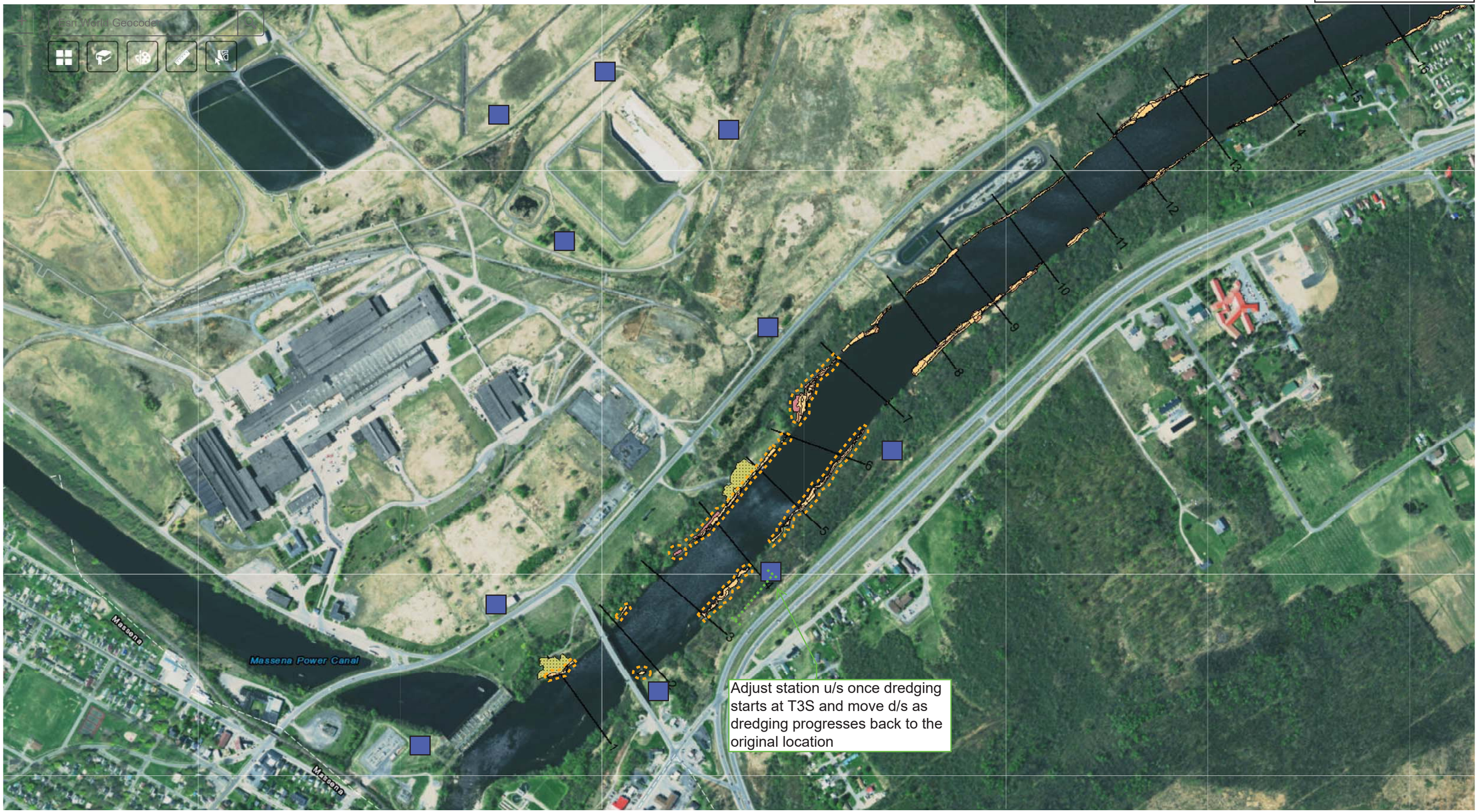
GENERAL VIEW SHOWING DMUs, FP REMOVAL AREAS, AND RECEPTORS

0.2mi
-74.838 44.987 Degrees

N

FIGURE 3

Grasse River Arconic Portal



600ft

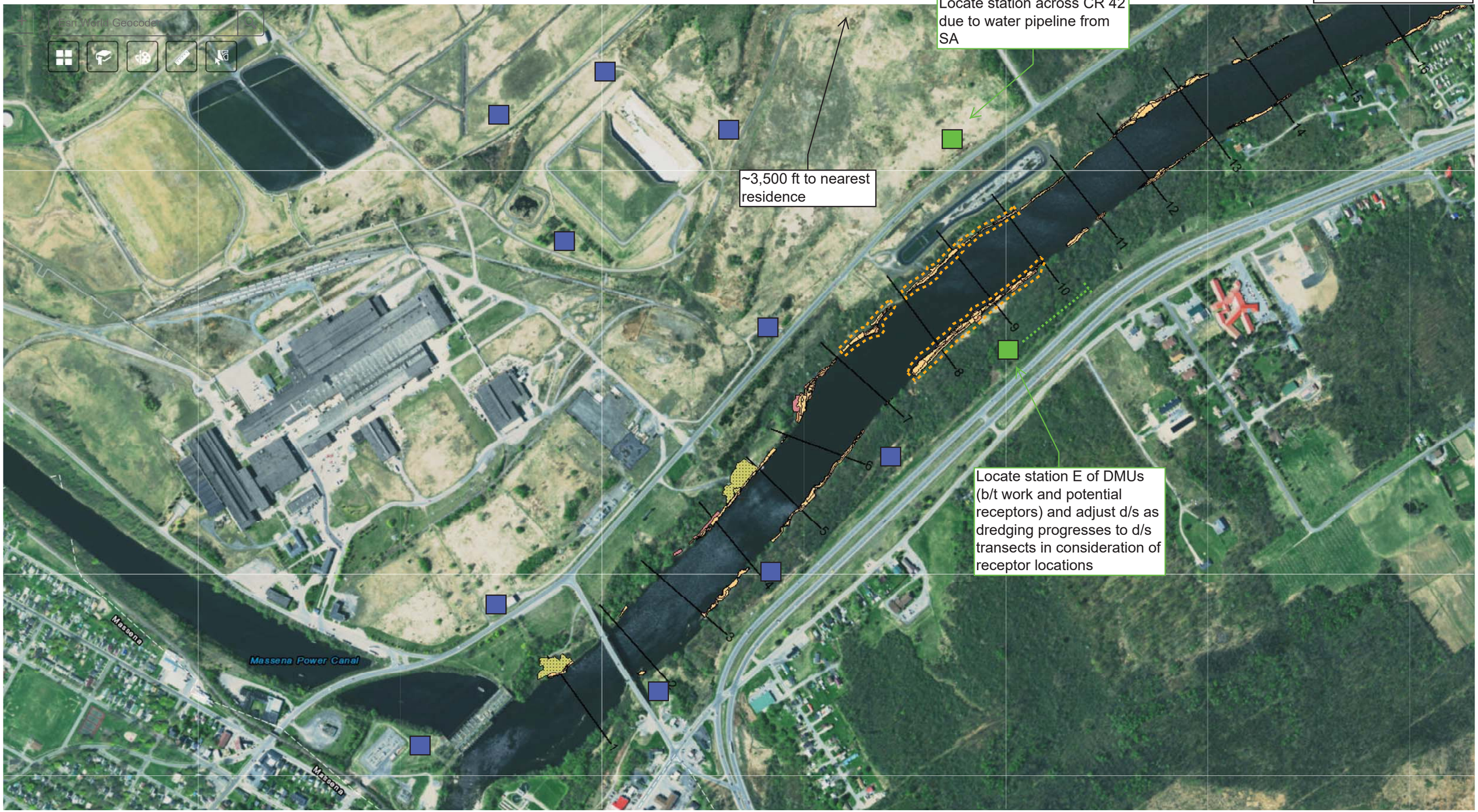
Click the map to get coordinates

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 4

Grasse River Arconic Portal



600ft

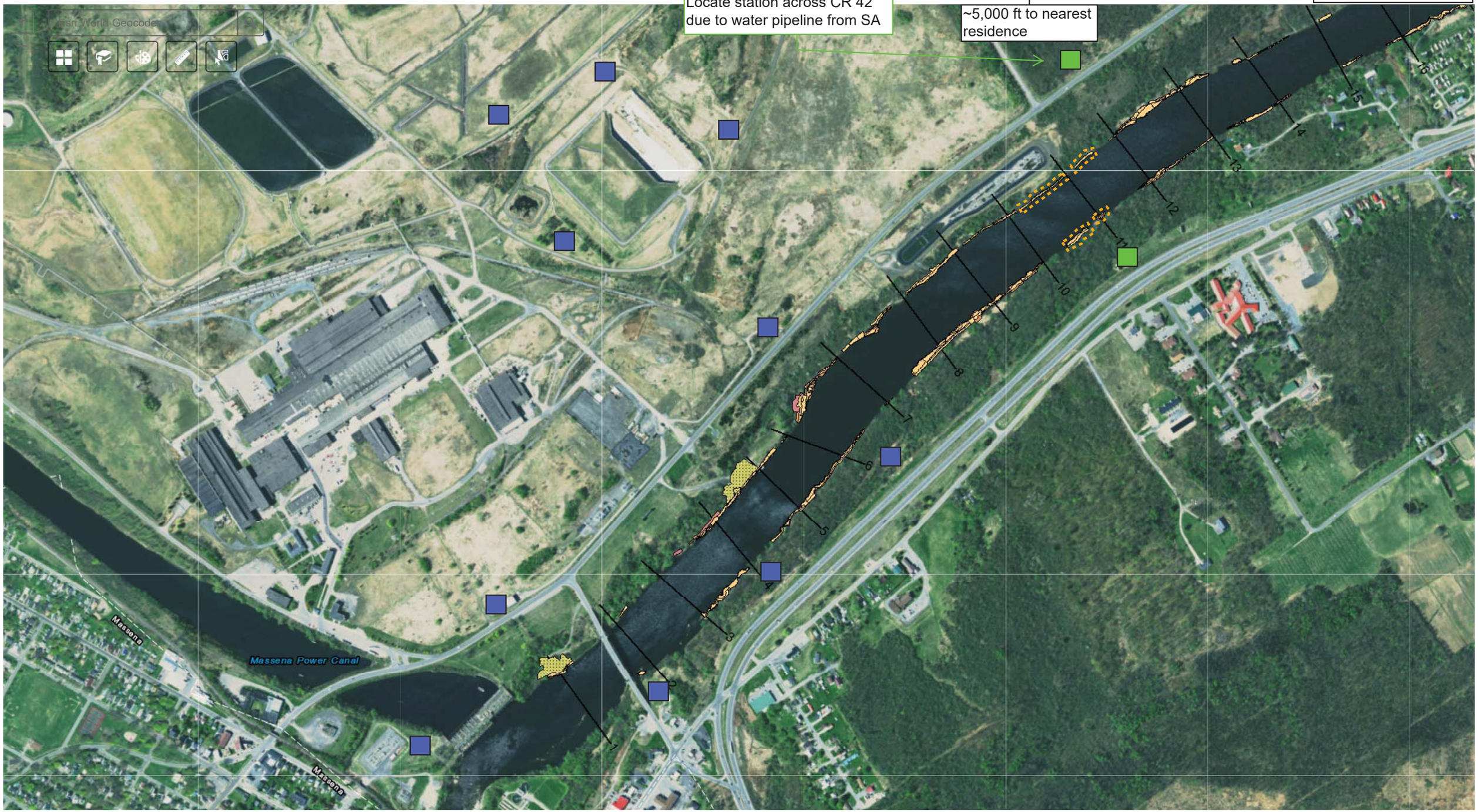
Click the map to get coordinates

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 5

Grasse River Arconic Portal



600ft

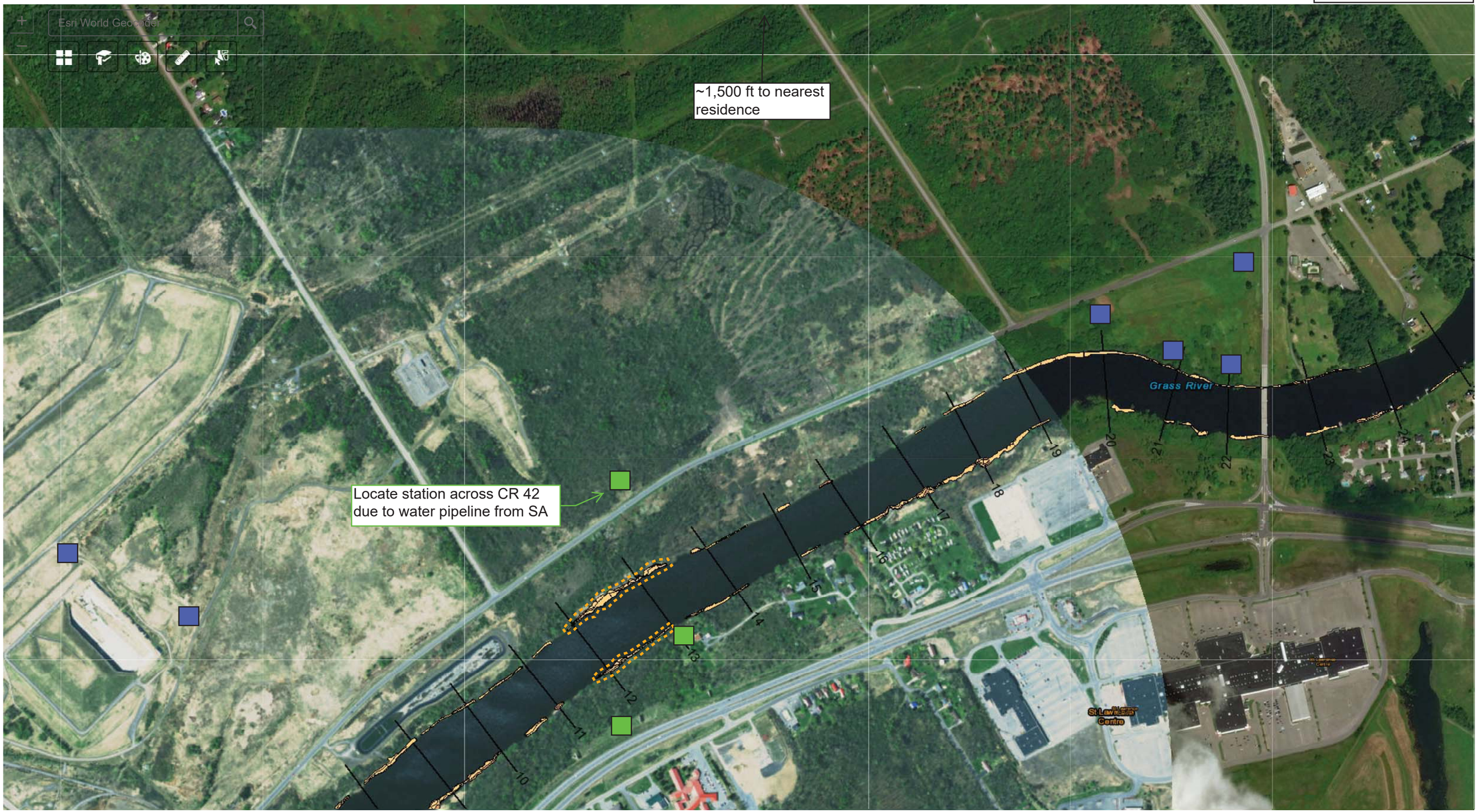
Click the map to get coordinates

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 6

Grasse River Arconic Portal



PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

600ft
-74.871 44.962 Degrees

N

FIGURE 7

Grasse River Arconic Portal

600ft
-74.871 44.962 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 8

Grasse River Arconic Portal

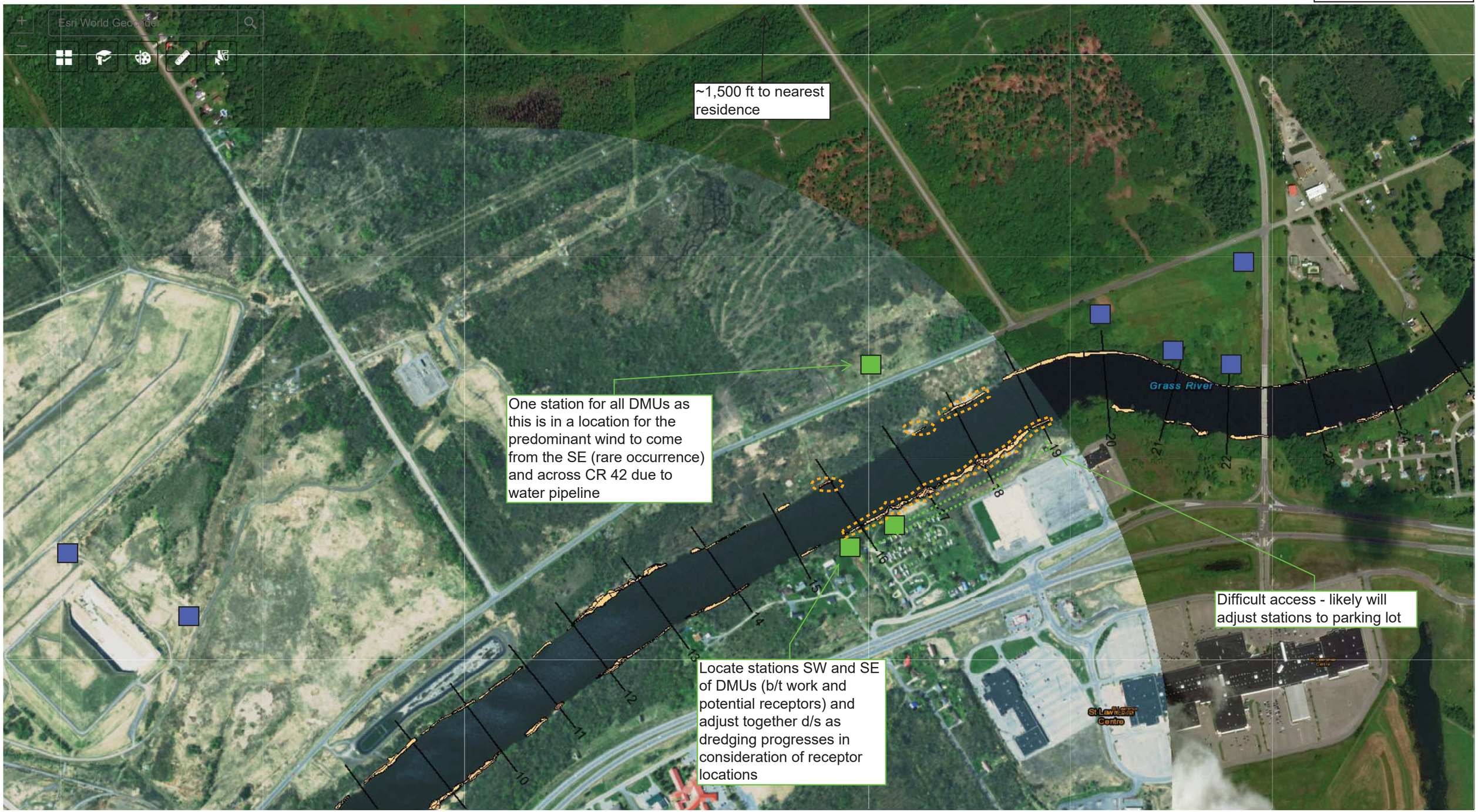
PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

600ft
-74.871 44.962 Degrees

N

FIGURE 9

Grasse River Arconic Portal



600ft
-74.871 44.962 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 10

Grasse River Arconic Portal

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

600ft
-74.871 44.962 Degrees

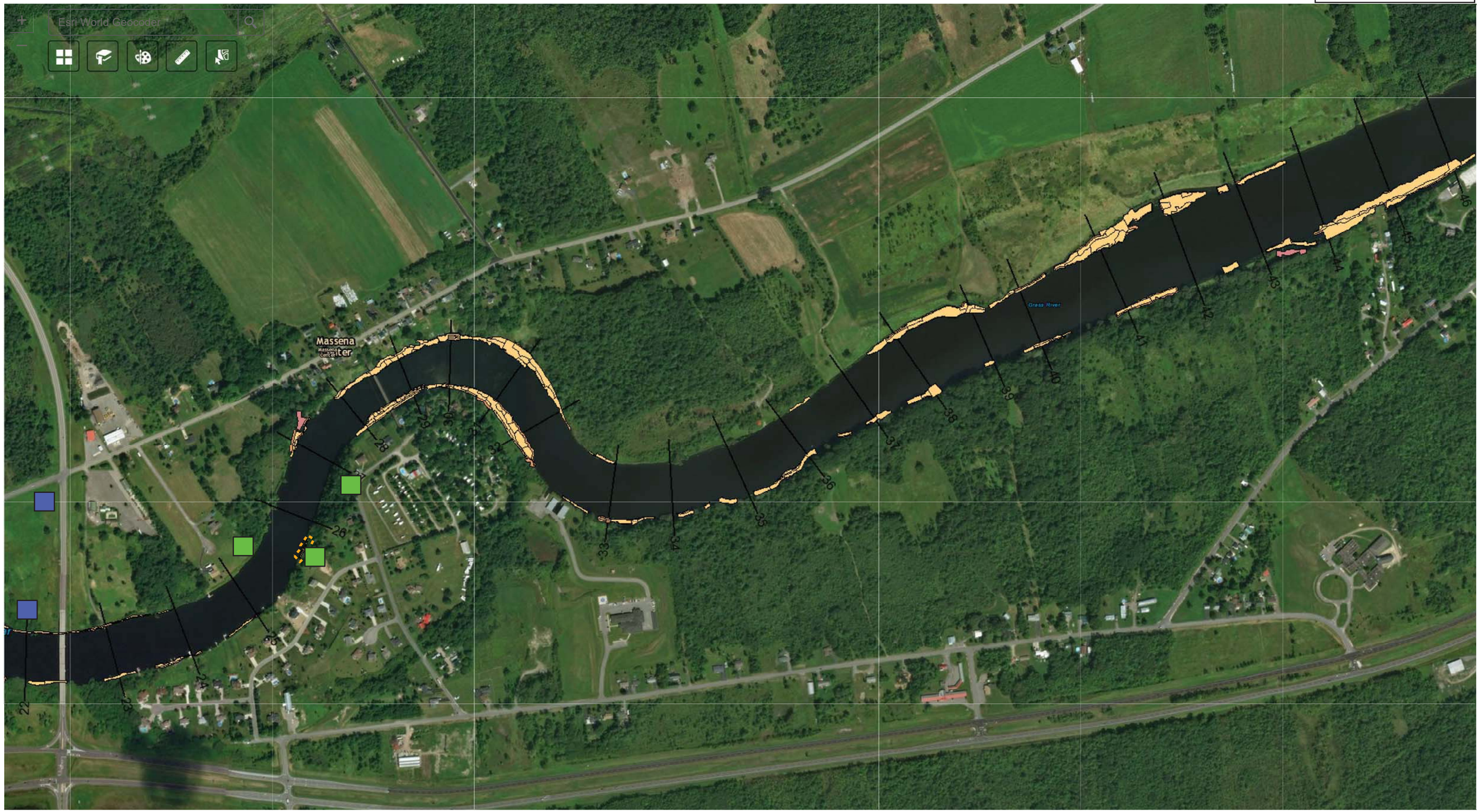
N

FIGURE 11

Grasse River Arconic Portal

Grasse River Arconic Portal

FIGURE 12



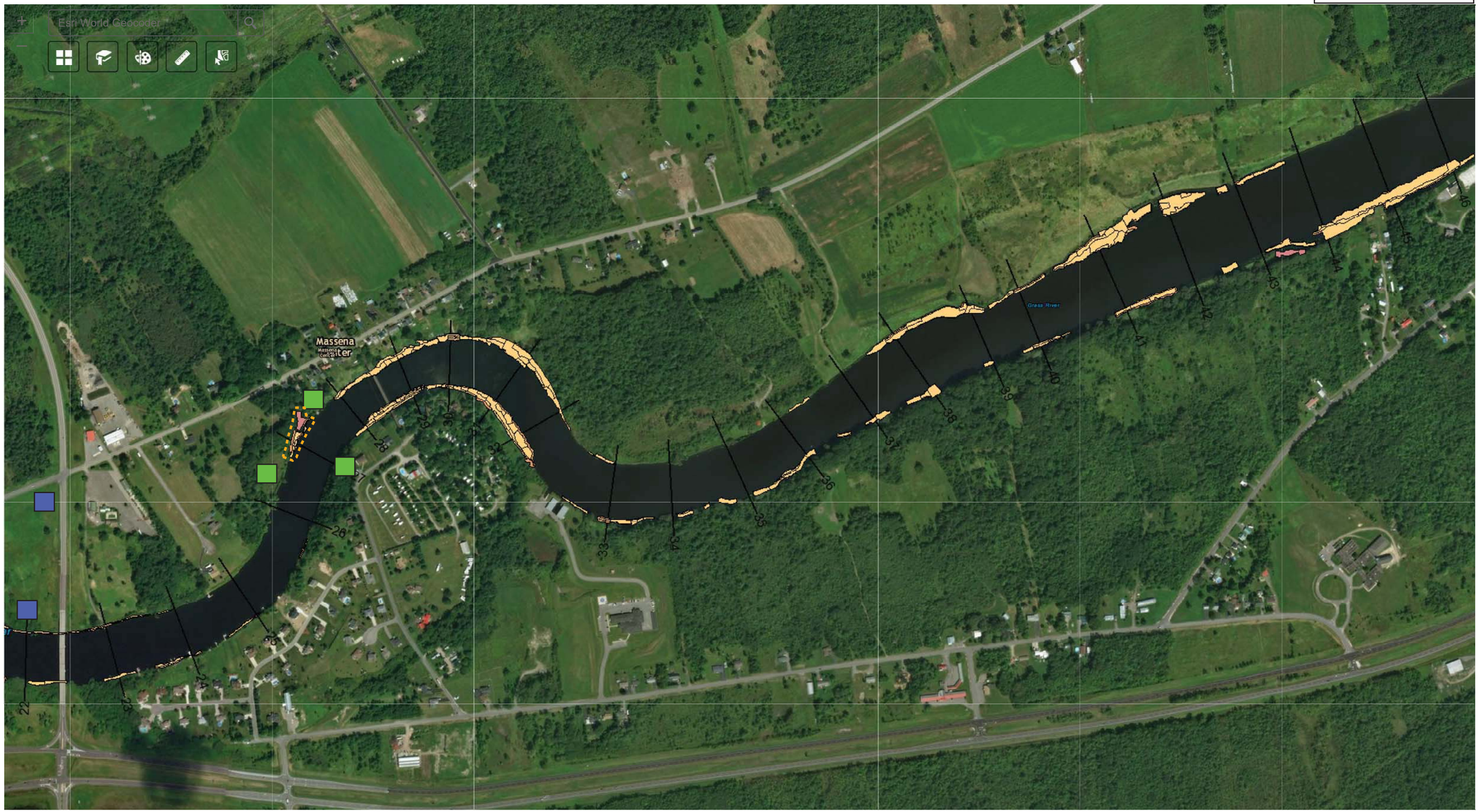
PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

600ft
-74.828 44.966 Degrees

N

Grasse River Arconic Portal

FIGURE 13



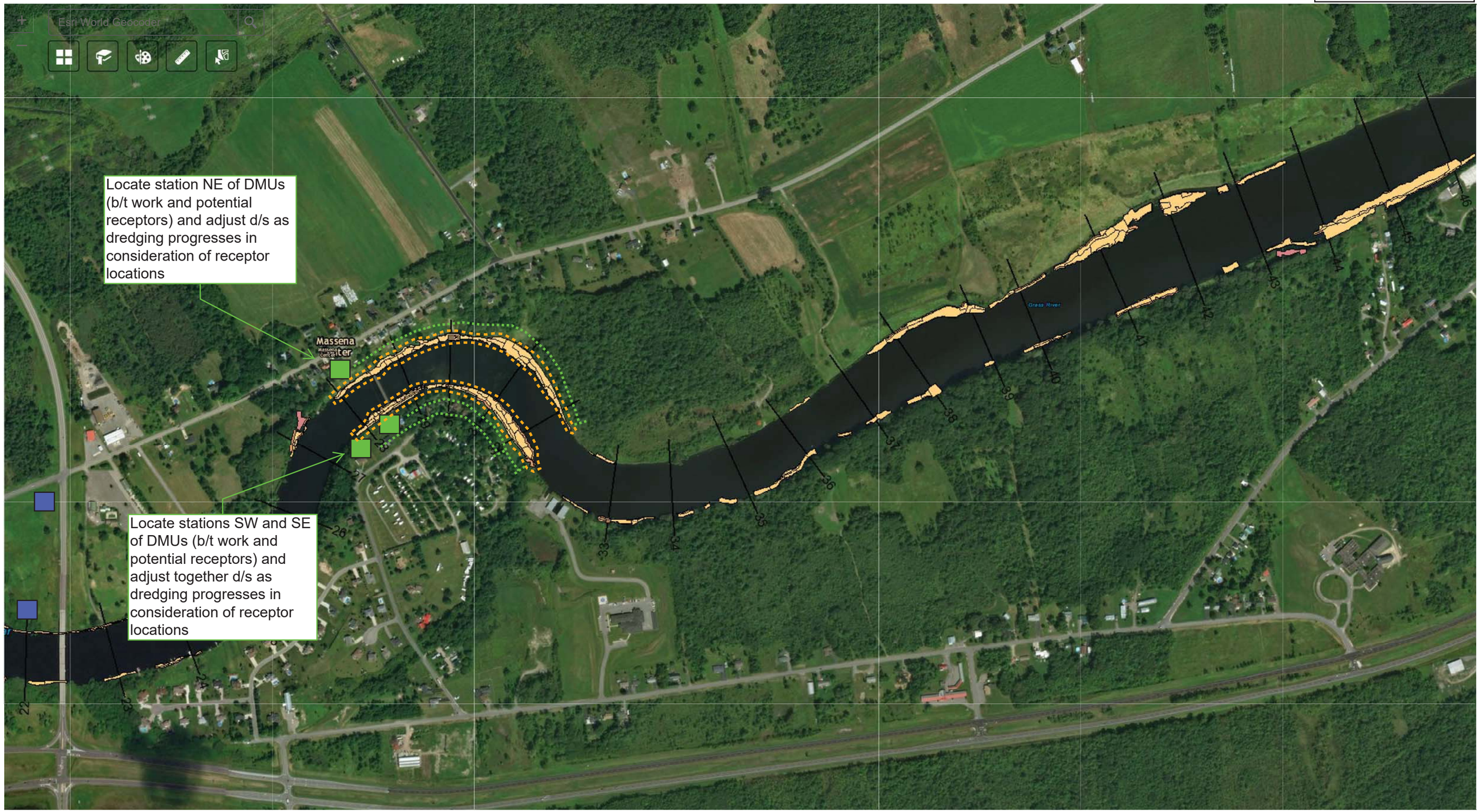
600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

Grasse River Arconic Portal

FIGURE 14



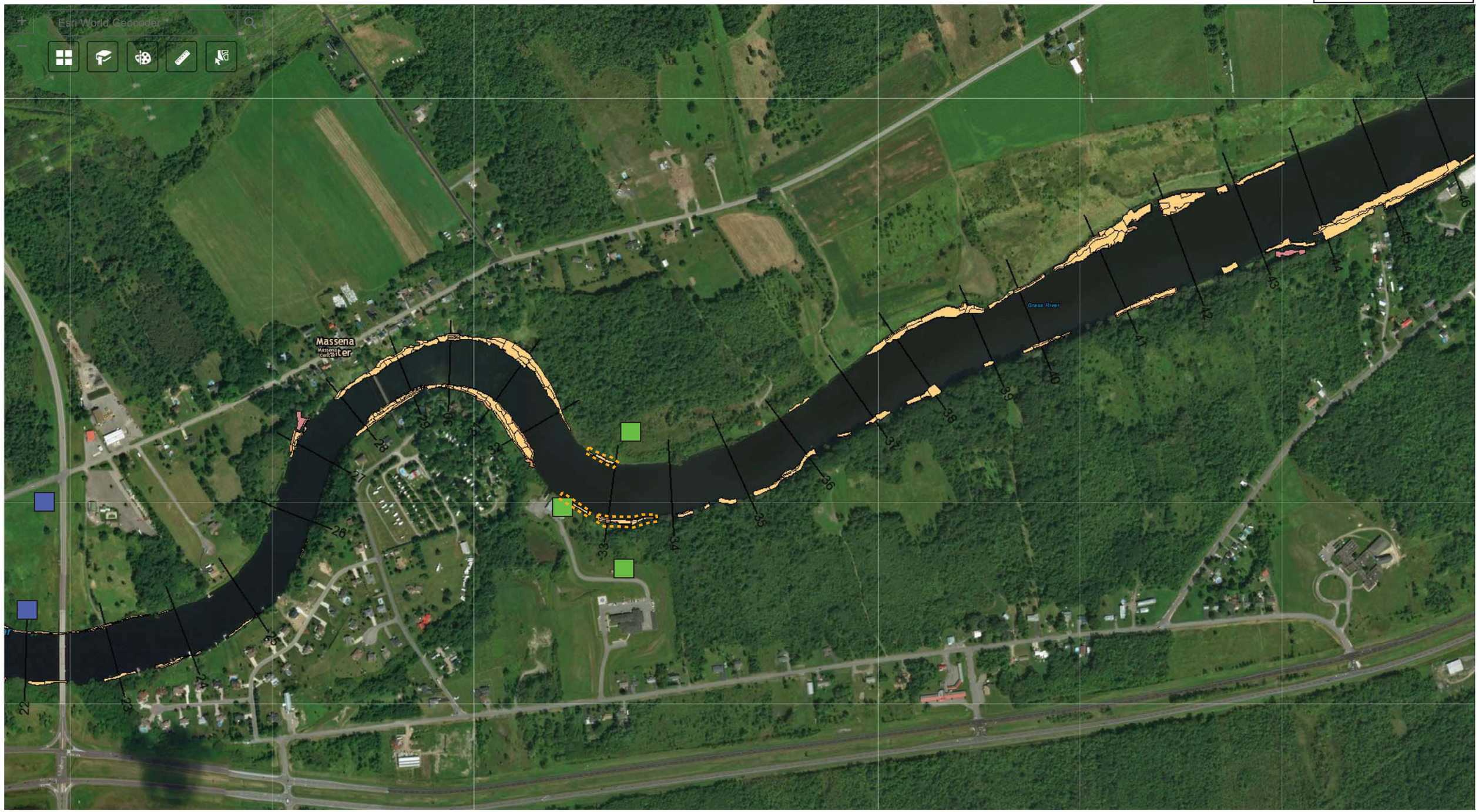
600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

Grasse River Arconic Portal

FIGURE 15



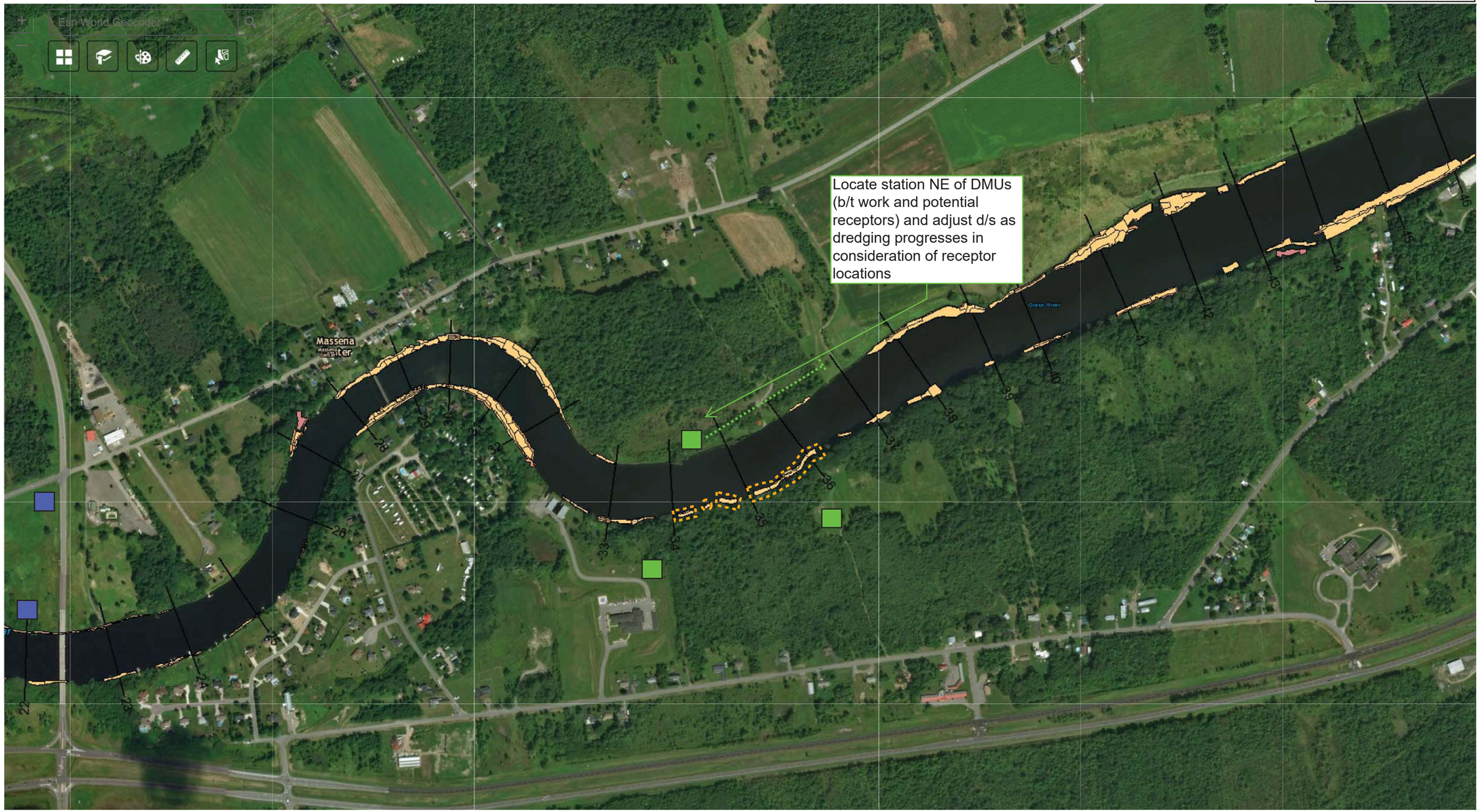
600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

Grasse River Arconic Portal

FIGURE 16



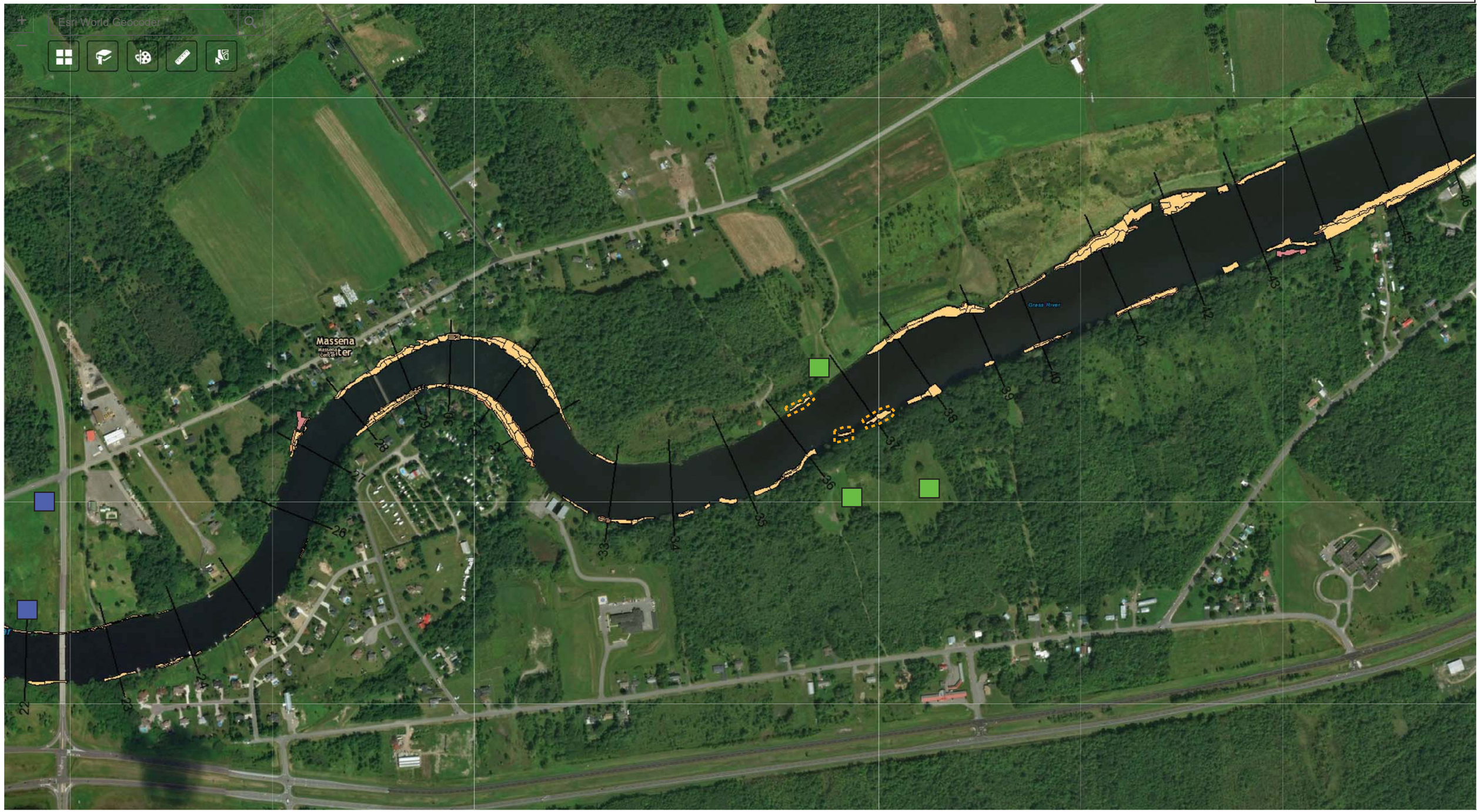
600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

Grasse River Arconic Portal

FIGURE 17



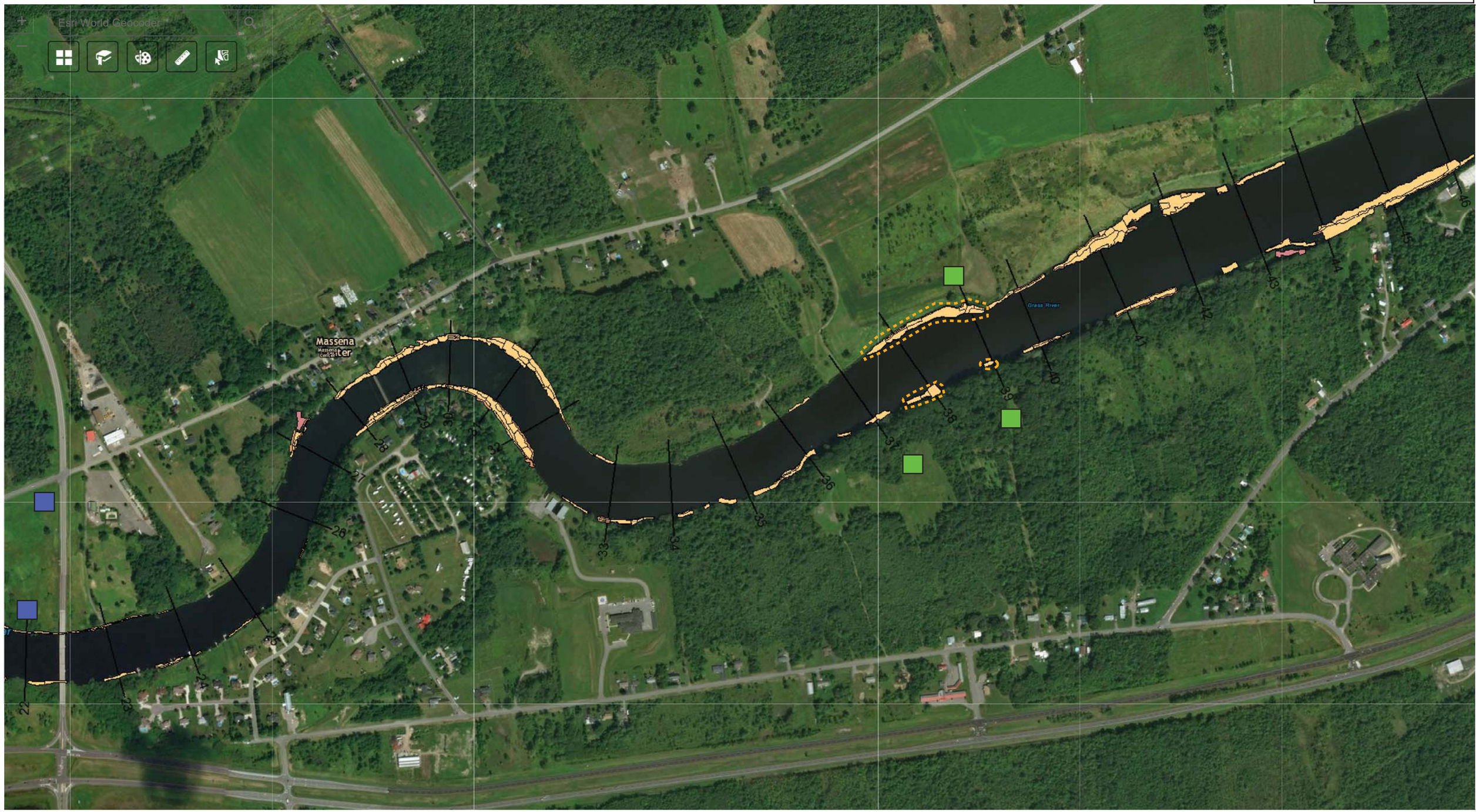
600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

Grasse River Arconic Portal

FIGURE 18



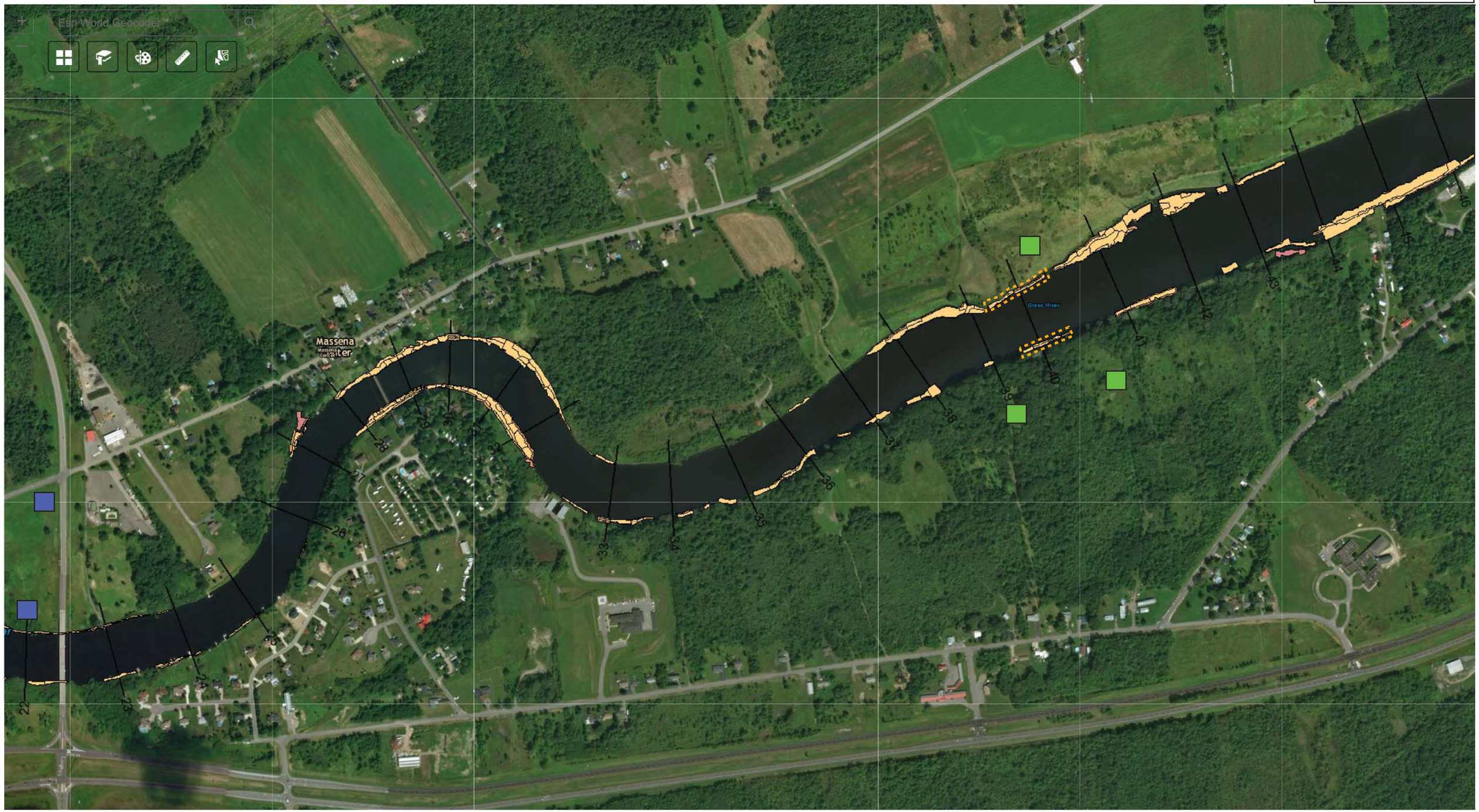
600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

Grasse River Arconic Portal

FIGURE 19



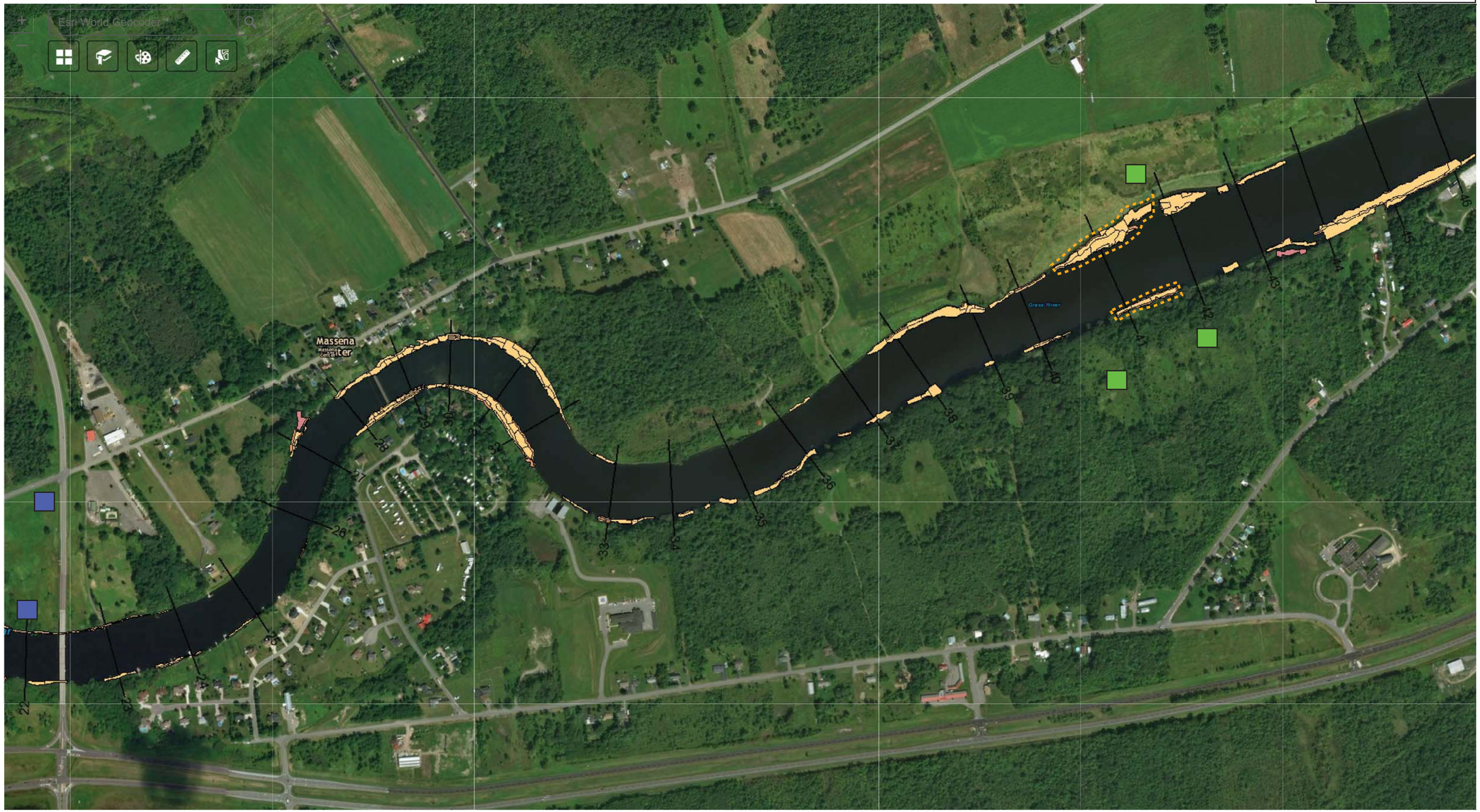
600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

Grasse River Arconic Portal

FIGURE 20



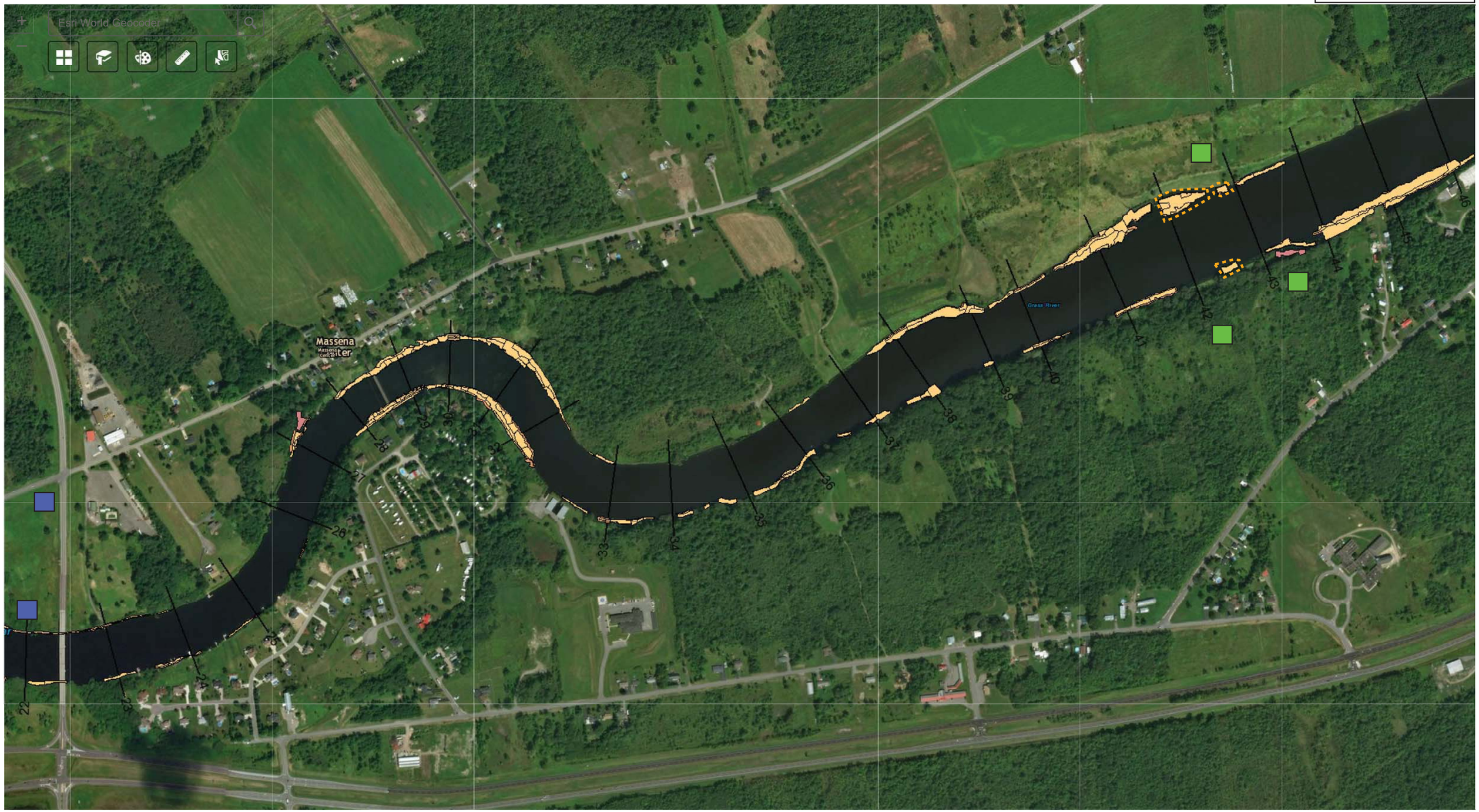
600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

Grasse River Arconic Portal

FIGURE 21



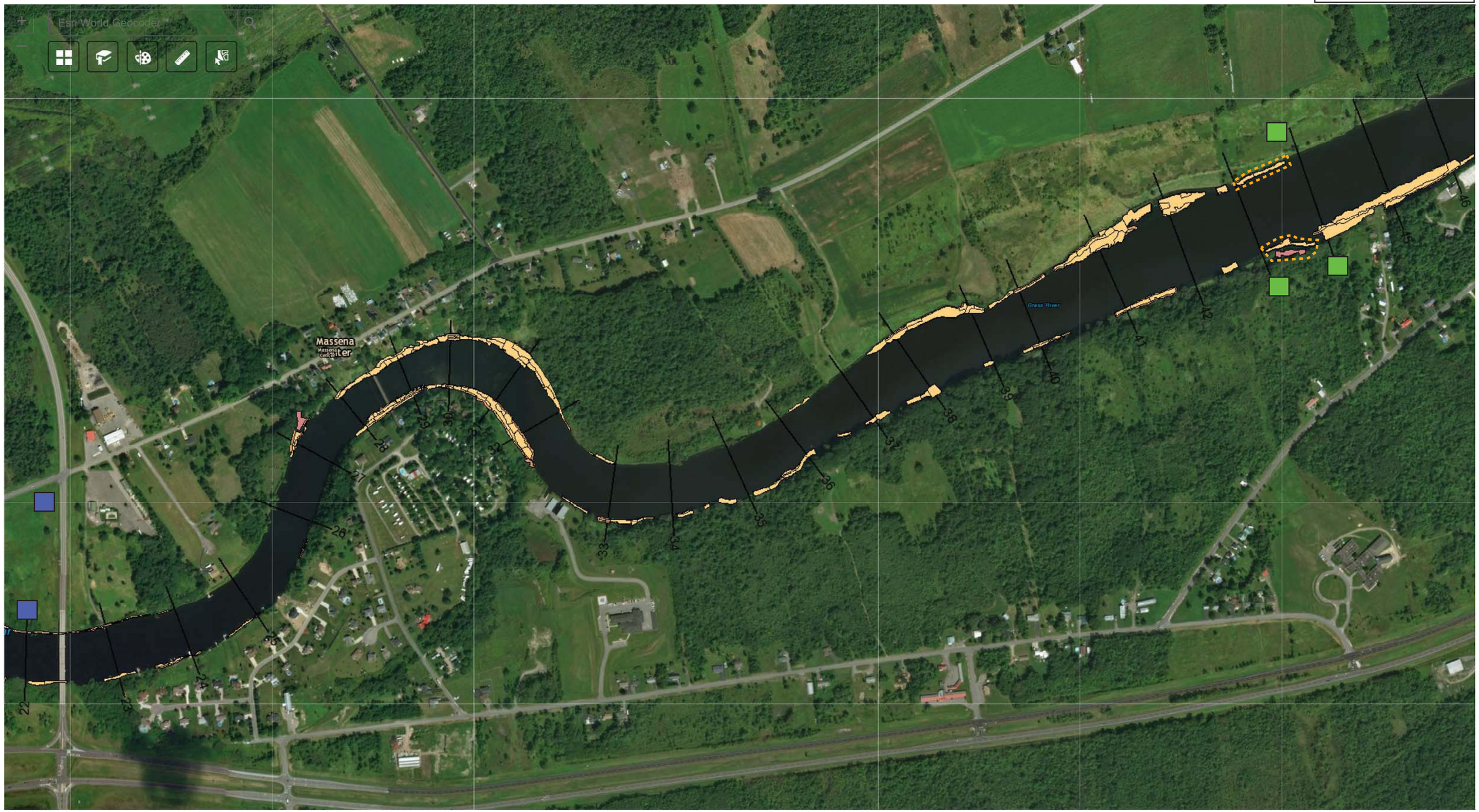
600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

Grasse River Arconic Portal

FIGURE 22



600ft
-74.828 44.966 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 23

Grasse River Arconic Portal

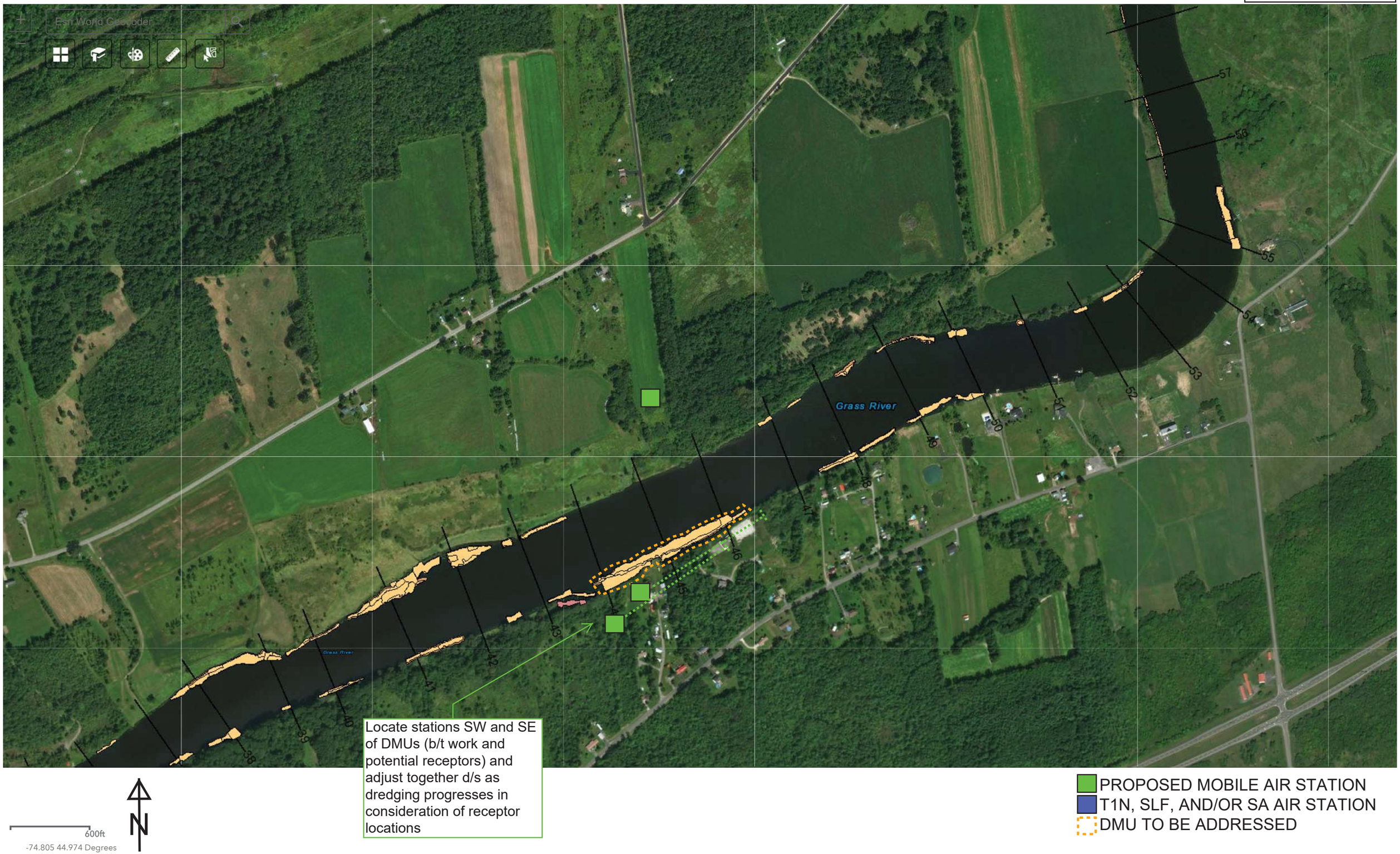


FIGURE 24

Grasse River Arconic Portal

FIGURE 25

Grasse River Arconic Portal

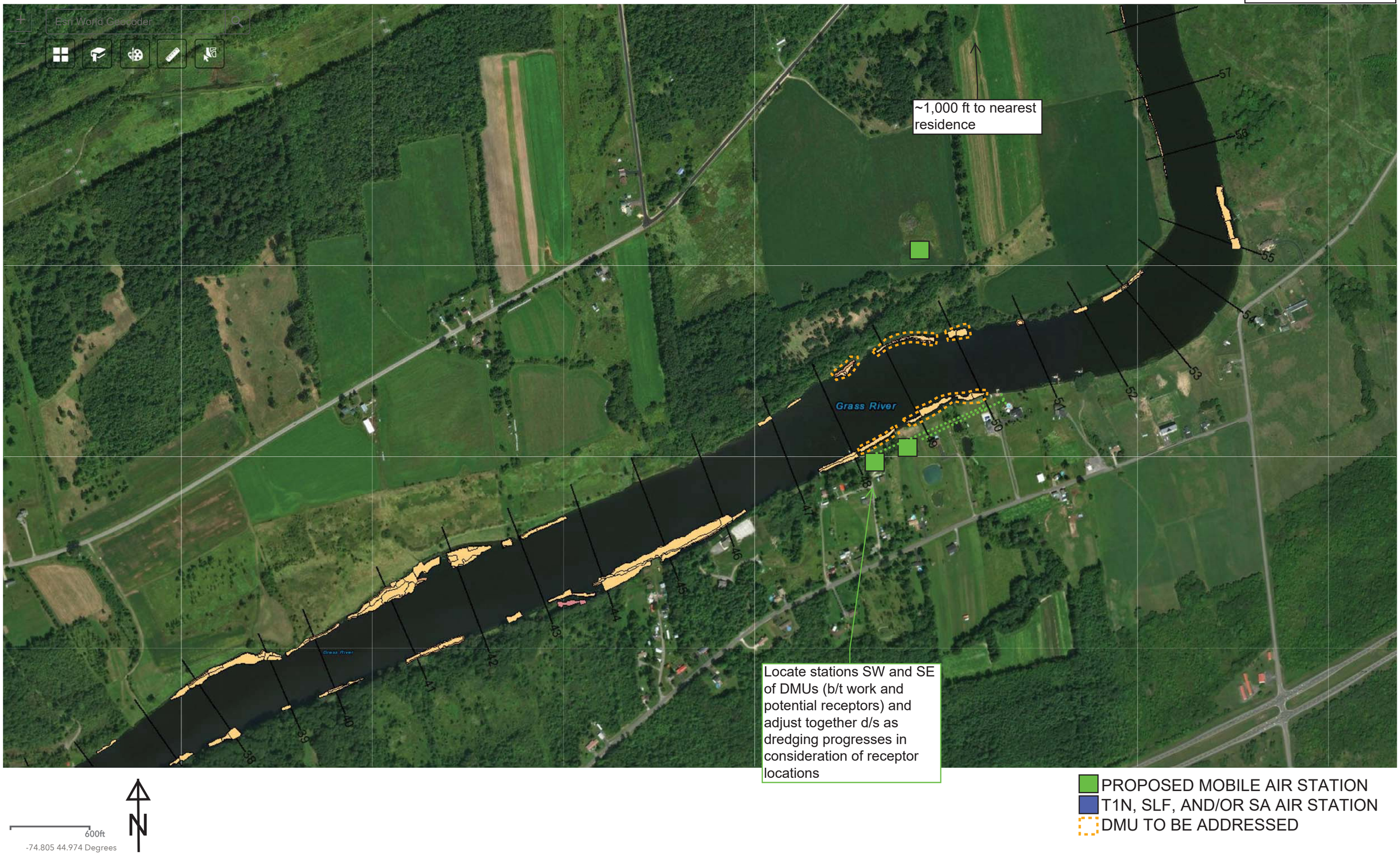


FIGURE 26

Grasse River Arconic Portal

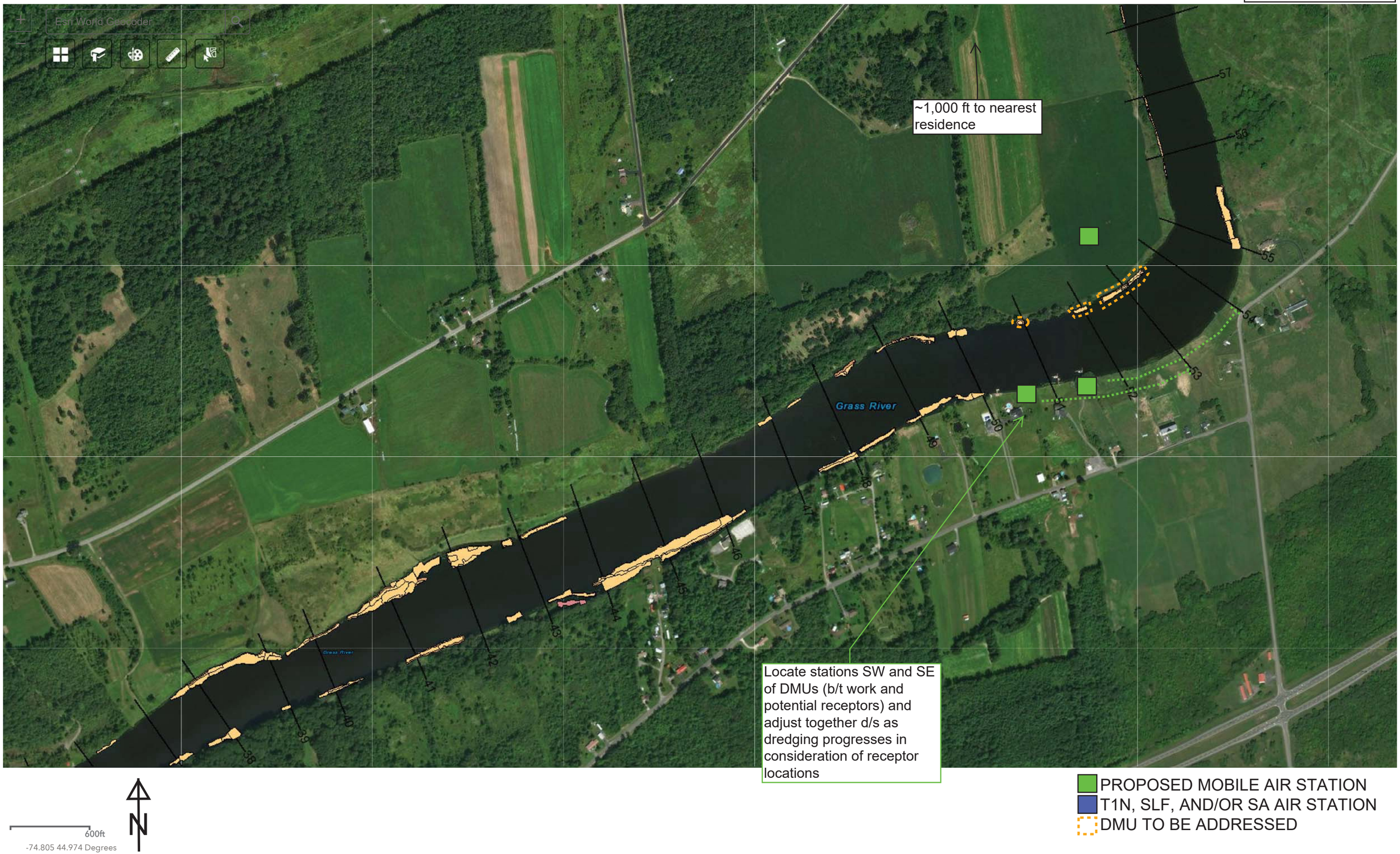
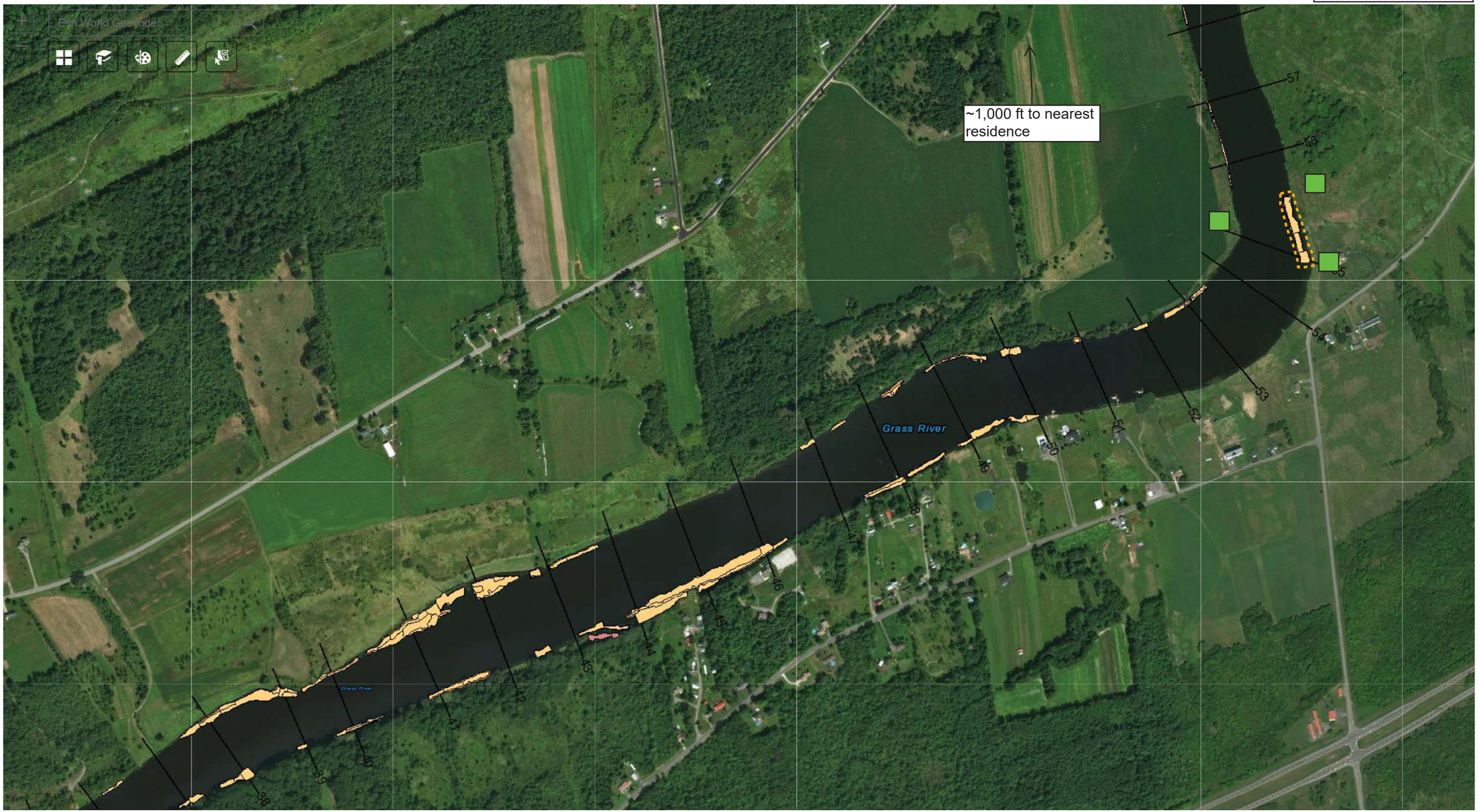


FIGURE 27

Grasse River Arconic Portal



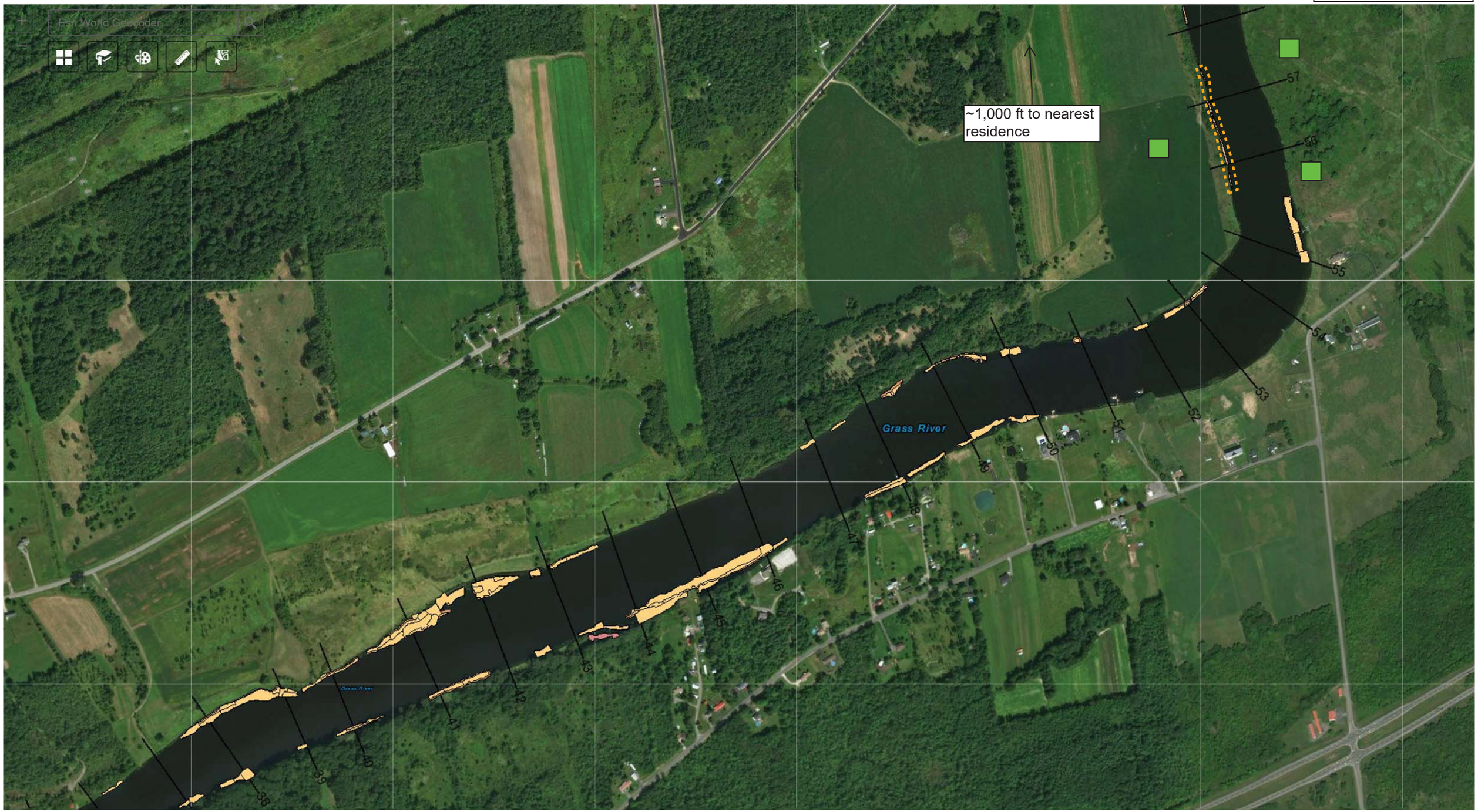
600ft
-74.805 44.974 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 28

Grasse River Arconic Portal



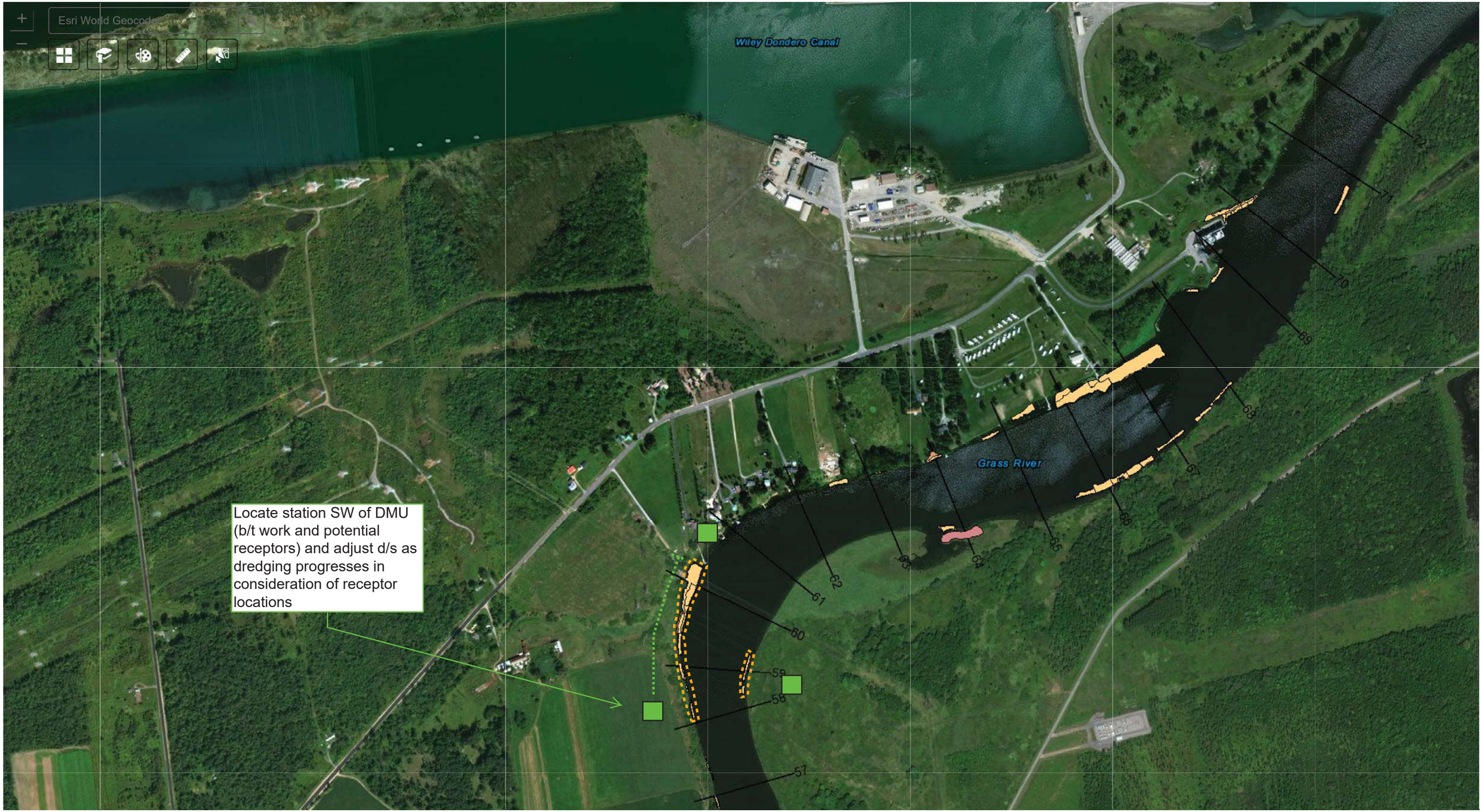
600ft
-74.805 44.974 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 29

Grasse River Arconic Portal



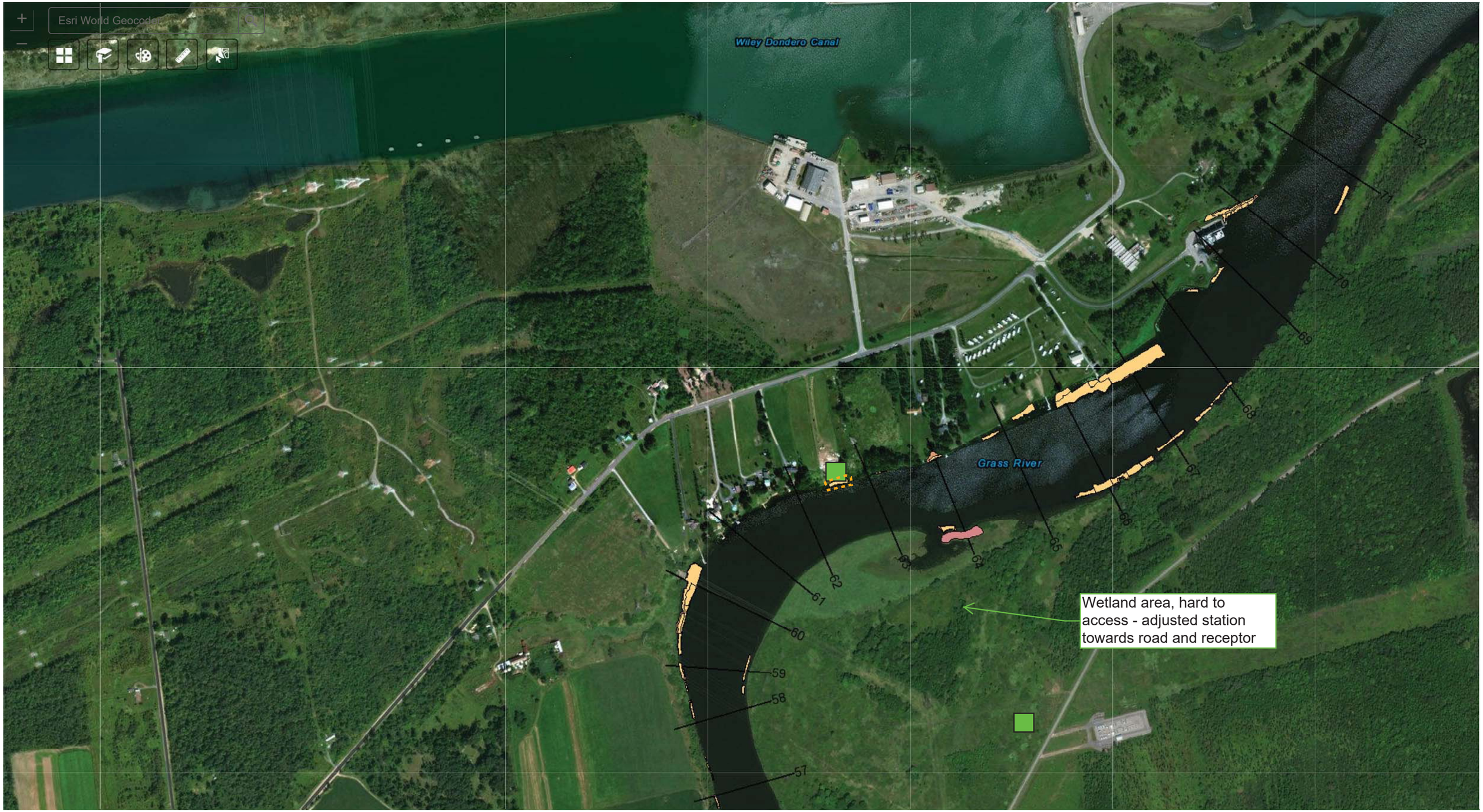
600ft
-74.804 44.986 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 30

Grasse River Arconic Portal



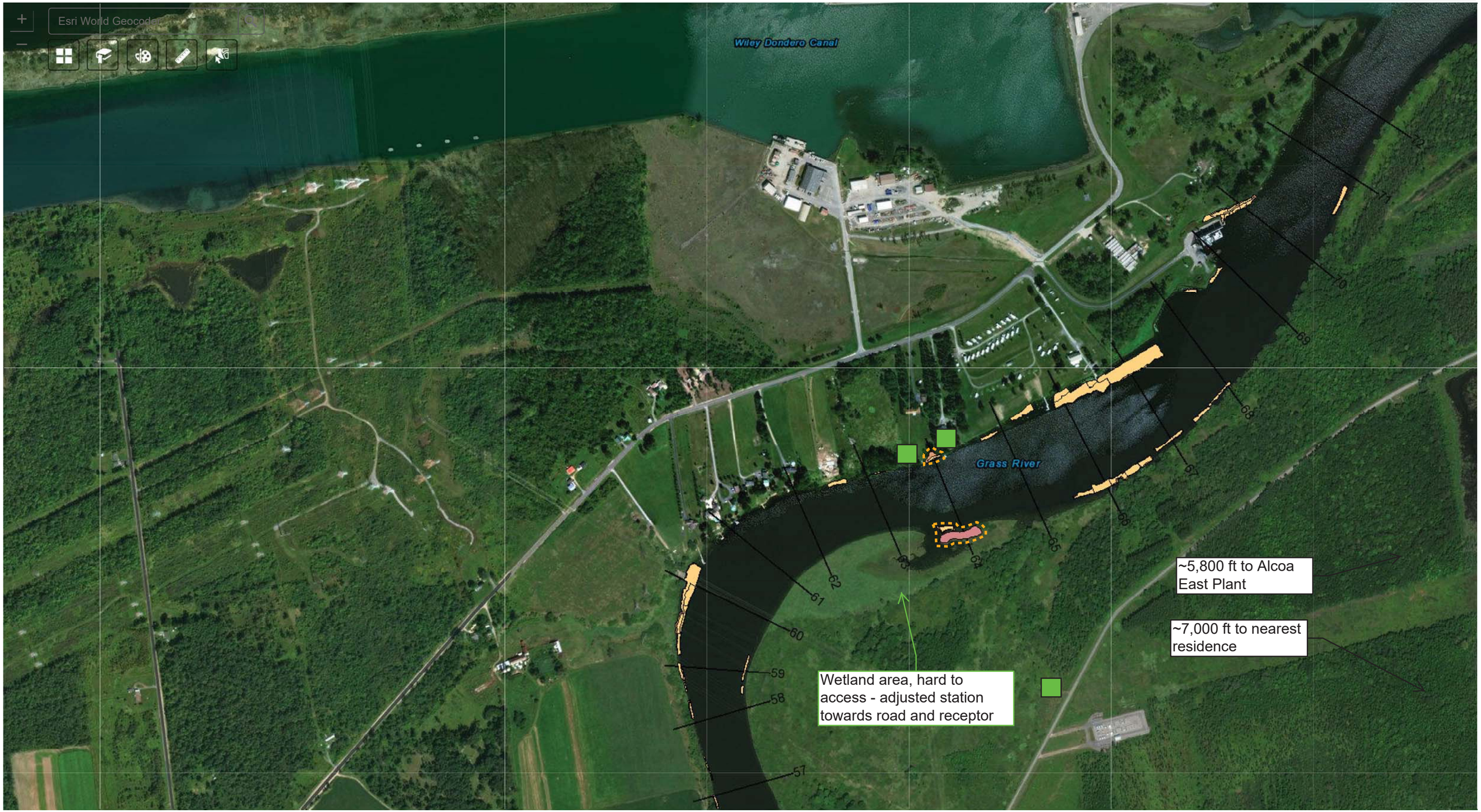
600ft
-74.804 44.986 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 31

Grasse River Arconic Portal



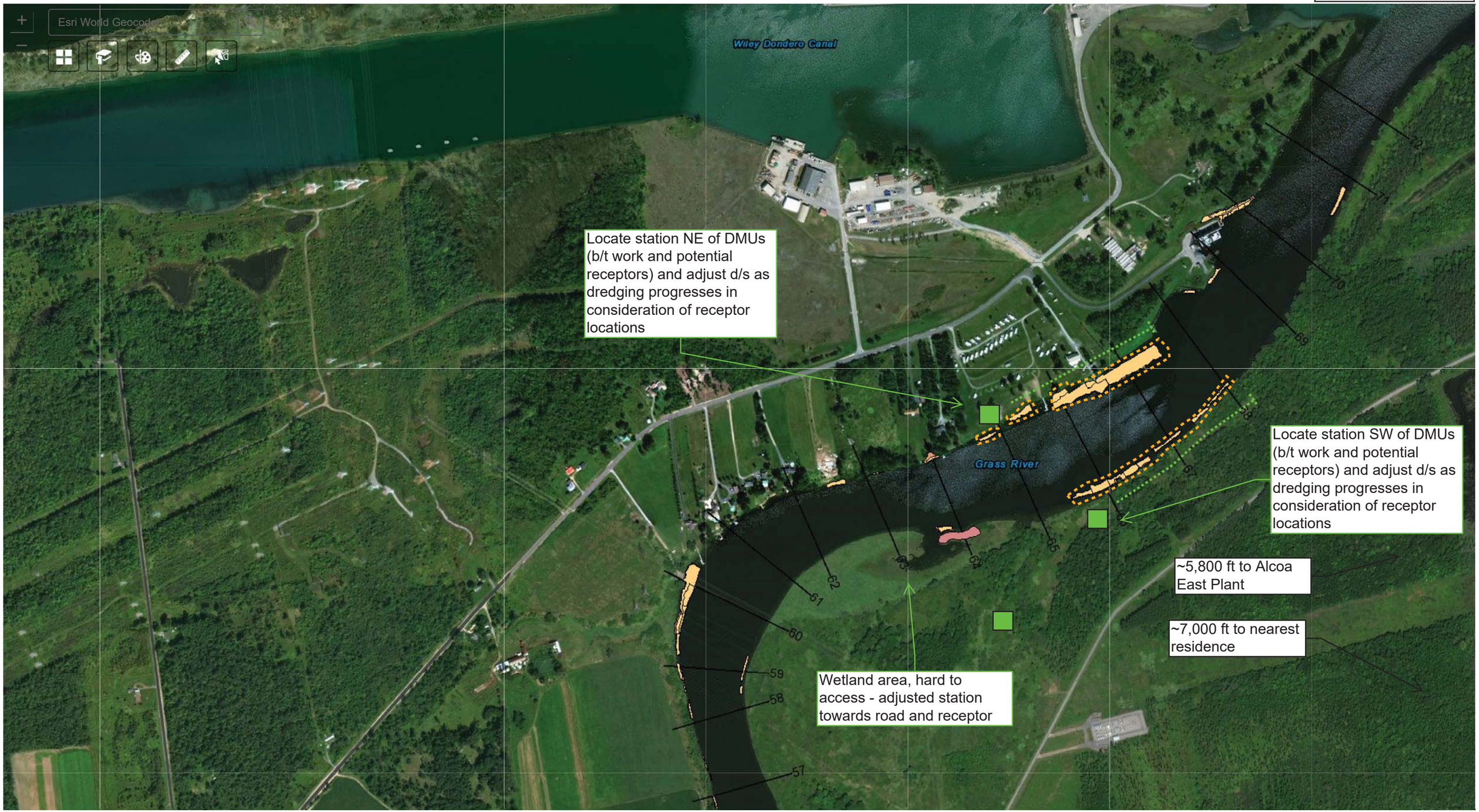
600ft
-74.804 44.986 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 32

Grasse River Arconic Portal



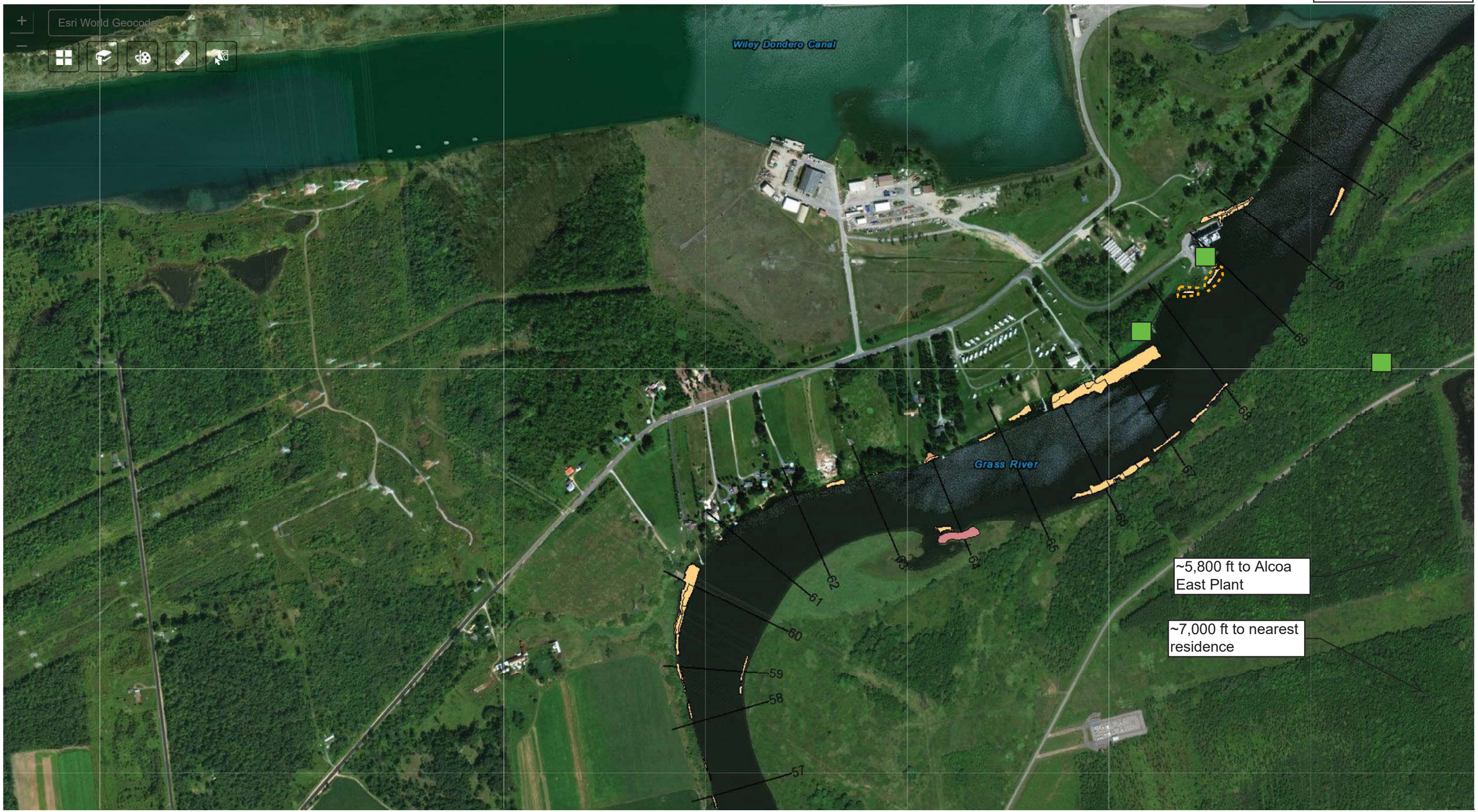
600ft
-74.804 44.986 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 33

Grasse River Arconic Portal



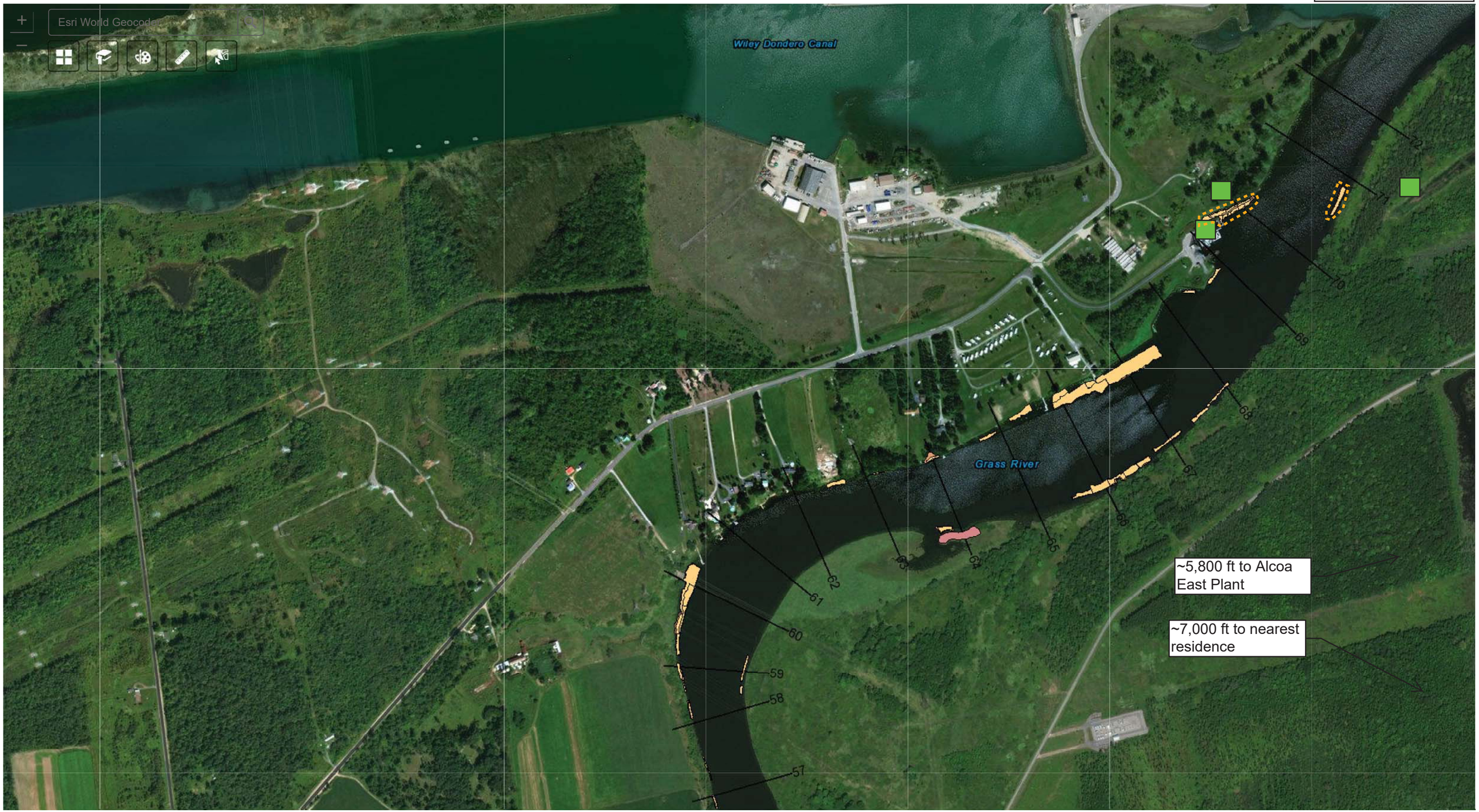
600ft
-74.804 44.986 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED

FIGURE 34

Grasse River Arconic Portal



600ft
-74.804 44.986 Degrees

N

PROPOSED MOBILE AIR STATION
T1N, SLF, AND/OR SA AIR STATION
DMU TO BE ADDRESSED