LOWER PASSAIC RIVER
ENVIRONMENTAL DREDGING PILOT STUDY
QUALITY ASSURANCE PROJECT PLAN
FINAL

LOWER PASSAIC RIVER RESTORATION PROJECT

November 21, 2005
(Revision 2)

Lower Passaic River
Environmental Dredging Pilot Study
Quality Assurance Project Plan
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Name	Agency or Company
Lisa Baron	NJDOT-OMR
Alice Yeh	EPA Region 2
Eric Stern	EPA Region 2
Scott Nicholson	USACE-NYD
Mike Thiagaram	TAMS/ET
Maheyar Bilimoria	TAMS/ET
Allen Burton	TAMS/ET
Ed Garvey	MPI
Scott Thompson	MPI
Timothy Wilson	USGS
Robert Chant	Rutgers University
Donald Hayes	Consultant
Michael Palermo	Mike Palermo Consulting
James Nickels	ASI
Reyhan Mehran	NOAA
Tim Kubiak	USFWS
Dave Risilia/Anne Hayton	NJDEP
Project File 72013	TAMS/ET

This Quality Assurance Project Plan (QAPP) for the Lower Passaic River Environmental Dredging Pilot Study is formatted in accordance with USEPA guidance (EPA QA/R-5 and QA/G-5). As noted in the table of contents, the cover page, table of contents, and distribution list comprise sections A1, A2, and A3, respectively. Therefore, the text of Section A begins with Section A4, immediately below.

A4 Project/Task Organization

An organization chart with the anticipated personnel is provided in Figure A-1. Details on the responsibilities of key personnel are presented below.

A4.1 Project Management

Agency Project Manager – Ms. Lisa Baron, NJDOT Office of Maritime Resources.

Ms. Baron's responsibilities include:
- Allocating agency resources to subcontractors (e.g., TAMS/ET, dredging contractor, etc)
- Coordination with, and point of contact for, other partner agencies including USEPA, USACE, USFWS, USGS, and NOAA
- Final authority for field decisions including modifications and changes

Project Manager – Dr. Maheyar R. Bilimoria (TAMS Consultants, Inc., *an Earth Tech Company* [TAMS/ET])

Responsibilities and duties of Dr. Bilimoria include the following:
- Define project objectives and establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task;
- Review and analyze overall task performance with respect to planned requirements and authorizations;
- Principal contact with, and assist the New Jersey Department of Transportation – Office of Maritime Resources (NJDOT-OMR) Project Manager in providing oversight of the Dredging Contractor;
- Manage Team Subcontractors [Malcolm Pirnie, Inc. (MPI), Aqua Survey Inc. (ASI), and commercial laboratories];
- Approve reports prior to their submission to NJDOT-OMR, United States Environmental Protection Agency (USEPA) Region 2, and United States Army Corps of Engineers (USACE); and
- Represent TAMS/ET and the project team at public meetings.

Technical Expert – Prof. Donald Hayes (Consultant)

Responsibilities and duties of Prof. Donald Hayes include the following:
- Advise the NJDOT-OMR Project Manager and TAMS/ET Project Manager regarding a variety of technical aspects of the Environmental Dredging Pilot Study and make recommendations to address the specific technical needs of the project;

- Review reports prior to their release to the general public; and
- Support the NJDOT-OMR Project Manager and TAMS/ET Project Manager as required.

Technical Expert – Dr. Michael Palermo (Mike Palermo Consulting)

Responsibilities and duties of Dr. Michael Palermo include the following:
- Advise the NJDOT-OMR Project Manager and TAMS/ET Project Manager regarding a variety of technical aspects of the Environmental Dredging Pilot Study and make recommendations to address the specific technical needs of the project;
- Review reports prior to their release to the general public; and
- Support the NJDOT-OMR Project Manager and TAMS/ET Project Manager as required.

Technical Advisor (Estuarine Dynamics) – Prof. Robert Chant (Rutgers University)

Responsibilities and duties of Prof. Chant include the following:
- Assist the NJDOT-OMR Project Manager and TAMS/ET Project Manager in developing and implementing the resuspension monitoring and sampling plan;
- Lead the project team from Rutgers University;
- Prepare and deploy fixed moorings and associated instruments (OBS, CDT, LISST, ADCP);
- Lead the hydrodynamic data collection effort using shipboard surveys (L and M boats) and fixed moorings and associated instruments (OBS, CDT, LISST, ADCP); and
- Download field data and reduce data for use by project team at conclusion of field event.

Technical Advisor (Resuspension Monitoring and Sampling) – Dr. Timothy Wilson (United States Geological Survey [USGS])

Responsibilities and duties of Dr. Wilson include the following:
- Assist the NJDOT-OMR Project Manager and TAMS/ET Project Manager in developing and implementing the resuspension monitoring and sampling plan;
- Lead the project team from USGS;
- Lead the water quality sample collection effort using shipboard surveys (TOPS boats) and associated instruments (Trace Organic Platform Sampler and ISCO sampler); and
- Coordinate with USGS laboratories and (in association with the QA Program manager) contract laboratory(ies) (e.g., STL) for sample submission and analysis.

Technical Advisor (Resuspension Monitoring and Sampling) – Dr. Edward Garvey (MPI)

Responsibilities and duties of Dr. Garvey include the following:

- Assist the NJDOT-OMR Project Manager and TAMS/ET Project Manager in defining technical project objectives related to the resuspension monitoring program and recommend procedures to address the specific technical needs of the project; and
- Support the NJDOT-OMR Project Manager and TAMS/ET Project Manager as required.

<u>Subcontractor Project Manager – Mr. Scott Thompson (MPI)</u>

The MPI Project Manager is directly responsible for activities performed by MPI personnel associated with the project. Other responsibilities include:

- Provide overall direction and management of MPI activities as defined in the QAPP;
- Provide QA management of aspects of the project within the responsibility of MPI; and
- Perform final review of all documents prepared by MPI.

<u>QA Program Manager – Allen Burton (TAMS/ET)</u>

The QA Program Manager will oversee the quality assurance aspects of the project. Specific responsibilities include:

- Preparation of the QAPP;
- Review data quality objectives, set assessment criteria and conduct assessments to determine compliance;
- Identification of appropriate analytical methodologies to generate the data to achieve the data quality objectives (DQOs);
- Establish the documentation, tracking, and sample shipping requirements for the various commercial/USGS and USEPA laboratories (DESA and CLP) receiving samples, and (in association with the site sample coordinator) verify that the field team is adequately trained and familiar with these requirements;
- Oversight of data verification and validation (as the current scope calls for USEPA to be responsible for the data validation, this task will consist mostly of tracking and compiling the validation performed by USEPA's staff or contractor);
- Coordinate analytical laboratory schedules; and
- Maintain the QAPP.

<u>Field Team Leader – Paul Kareth (TAMS/ET)</u>

Field activities on the boat and on shore will be managed by the Overall Field Team Leader. Responsibilities include:

- Manage field staff;
- Supervise Site Coordinator and Sample Collection Coordinator;
- Coordinate sample collection and field sample processing schedules;
- Coordinate and manage the team subcontractors;
- Monitor program progress relative to schedule and determine corrective actions necessary to maintain schedule;

- Review/approve the type of field equipment used and verify that procedures are followed to achieve the DQOs;
- Review field notebooks/logs with respect to completeness, consistency, and accuracy; and
- Prepare routine progress reports, including a summary of field activities and field audit results.

Site Coordinator and Sample Collection Coordinator – Celeste Foster (TAMS/ET)

The Site Coordinators are responsible for day-to-day supervision of site activities. Specific responsibilities include:

- Primary contact with Agency oversight team;
- Oversee field laboratory activities, including field data log in, TR/COC generation (including Forms II Lite-generated documentation), sample labeling, cooler packing;
- Coordinate sample collection and field laboratory schedules; and
- Report deviations from protocol to the Field Team Leader.

Site Health and Safety Manager – Mr. F. Christopher Purkiss (MPI)

The Site Health and Safety Manager is responsible for establishing and monitoring compliance with the site Safe Work Plan (SWP) and OSHA standards (29 CFR 1910.120) for the overall field project. Specific responsibilities include:

- Establish and monitor compliance with procedures specified in the SWP during performance of field work activities;
- Conduct daily health and safety/subcontractor coordination meetings;
- Site decisions on health and safety issues (except for those under the jurisdiction of boat captains); and
- Report deviations from SWP to Project Manager.

Oversight Agencies

- USACE will provide engineering/construction oversight during the pilot dredging program
- USEPA Region 2 and NJDEP will provide overall program oversight.

A4.2 Project Execution

Pilot Dredging Operations (Contractor, Jay Cashman, Inc., selected through NJDOT bid procurement process)

Responsibilities and duties of Dredging Contractor include the following:

- Comply with all of the requirements of the contract with NJDOT-OMR
- Perform a daily check of the Real Time Kinematic - Differential Global Positioning System (RTK-DGPS) by checking a point with known coordinates;
- Dredge 5,000 cubic yards of sediment from the target dredge prism;

- Place dredged sediment into barges/scows; and
- Transport barges/scows to sediment decontamination and offloading facility (Bayshore Recycling).

Sample Collection
TAMS/ET, MPI, USGS, and Rutgers University

The field personnel conducting water sample collection (TOPS boats) have the following responsibilities:
- Initial acquisition and set-up of required sampling equipment (e.g., Trace Organics Platform Sampler and associated sample media including glass fiber filters and XAD resin; ISCO Samplers; etc.) and containers. It is anticipated that personnel experienced in the TOPS deployment (e.g., from USGS) will participate both in the planning and implementation of the TOPS sample collection.
- Collect water samples at pre-determined depths and locations and maintain field logs;
- Perform a daily check of the Global Positioning System (GPS) by checking a point with known coordinates; and
- Deliver field logs and samples to the staging area throughout each of five days in the field.

Sample Processing (R/V Hudson)
TAMS/ET and MPI

TAMS/ET and MPI will process the water samples (with limited assistance from ASI, if necessary). Specific responsibilities of the personnel processing samples include:

- Enter field notes into electronic database;
- Place samples into sample containers (e.g., coolers);
- Prepare sample containers for shipping; and
- Maintain chain of custody documentation.

Hydrographic Surveys
TAMS/ET and ASI

TAMS/ET will provide oversight of ASI staff conducting pre- and post-dredging hydrographic surveys. ASI responsibilities include:
- Conduct hydrographic surveys at the project site in the Harrison Reach of the Lower Passaic River
- Conduct tests of surveying equipment according to the manufacturers' guidelines
- Maintain field logs; and
- Perform a daily check of the RTK-DGPS by checking a point with known coordinates.

Project QA/QC Officer [MPI/ET; TBD; may or may not be the QA Program Manager]

The Project QA/QC officer has the following responsibilities:

- Receipt of data packages from laboratories;
- Review laboratory data packages;
- Coordinate field QA/QC activities with Field Team Leader;
- Review field reports;
- Review audit reports;
- Prepare interim Quality Assurance Reviews; and
- Prepare final Quality Assurance Reviews.

<u>Analytical Measurements on Water Quality and TSS Samples</u>

To the extent feasible, analytical work for this project will be arranged through USEPA-coordinated laboratories (e.g., CLP non-RAS [non-Routine Analytical Services] and the USEPA Region 2 DESA laboratory in Edison, NJ). Commercial laboratories (contracted by MPI or TAMS/ET under NJDOT-OMR contract) may be utilized as needed to perform specialized analyses beyond the scope or capacity of either DESA or CLP laboratories. Additional support, including filtering of samples and preparation of filters for particulate organic carbon, will be provided by the USGS laboratory in Trenton, NJ.

The specific analyses that the analytical laboratories will perform are identified in Section B4. In general, it is anticipated that the analytical laboratories utilized will include the following (note that several different CLP laboratories may be used):

- USEPA Region 2 DESA Laboratory – Metals (except mercury). Analysis by CLP SOW ILM05.3, ICP-MS; except iron by ICP-optical emission spectroscopy (ICP-OES); total organic carbon on water (DOC by USEPA Region 2 SOP C-83) and on particulate matter (POC; Region 2 SOP C-88); Bromide/Chloride by USEPA Method 300.0; and Total Suspended Solids (Region 2 SOP C-33; based on USEPA method 160.2)
- Commercial laboratory (tentatively assigned to STL-North Canton, OH facility): mercury (analysis by USEPA Method 1631E); includes provision of specialty-cleaned 2.5-L bottles and tubing
- CLP non-Routine Analytical Services (non-RAS) – High resolution analyses. Dioxins/Furans (CLP DLM02.0) and PCB congeners (USEPA Method CBC01.0); and pesticides (CLP SOW SOM 01.1).

Responsibilities and duties of the analytical laboratories include the following:

- Perform the specified analytical procedures;
- Report the data to TAMS/ET (contract-required USEPA contact for CLP laboratories) and the database manager (MPI) in the required format and within required turnaround times; and
- Conformance with the protocols in the QAPP and specified analytical method, and contact the QA Program Manager (or USEPA RSCCC for EPA-arranged analyses) prior to any protocol deviations.

<u>Coordination of Analytical Laboratories</u>
TAMS/ET (for commercial analyses)
USEPA Region 2 Regional Sample Control Coordinator (RSCC) (for DESA, CLP or other USEPA-arranged analyses)

Responsibilities include:

- Assist in procurement of USEPA (CLP or DESA) laboratory services;
- Coordination with the analytical laboratories;
- Monitor the progress of the analytical work, including preliminary, electronic, and hard copy final deliverables; and
- Resolve laboratory questions/concerns regarding analytical or deliverable requirements.

<u>Data Production and Database Development and Maintenance</u>
TAMS/ET and MPI

TAMS/ET will provide oversight of the production of data tables and maps that will be prepared by MPI resulting from this field effort. MPI is the Database Manager for the overall Lower Passaic River Restoration Project. Responsibilities include:

- Electronic QA checks on data packages;
- Electronic data verification;
- Population of the project database;
- QA checks on database; and
- Distribution of database.

<u>Data Quality Review and Validation</u>

Validation of CLP and other analyses performed by or through USEPA will be performed by the Environmental Services Assistance Team (ESAT) (USEPA Region 2), Edison, NJ. USEPA Region 2 SOPs will be utilized for data validation (see further discussion in Section D.2 of this QAPP), regardless of whether the chemical analytical data analysis is performed by a CLP laboratory or a subcontract laboratory. There is no Region 2 SOP currently available for validation of PCB congener data (USEPA Method 1668A or CLP SOW CBC01.0); other available agency guidelines will be used for validation of those data (see D.2).

Analyses performed by the Region 2 DESA laboratory are validated internally and do not require third-party validation; this review is documented in the cover letter accompanying the data report.

Based on the data quality objectives, full validation is not necessary for the conventional-type analyses; rather, a less rigorous data quality review will be conducted for these analyses. Such review will include verification that the samples were analyzed by the requested method, and the important protocols specified by the method were followed (to the extent that the required deliverables allow such review). An individual familiar with the site and the method will also check that the reported results are reasonable, and method-required QC is within acceptable limits.

Responsibilities include:

- Validate data on a timely basis to facilitate incorporation of qualifiers into the database.
- Identification of any data which have limited usability or which are unusable for their intended purpose.
- Provide recommendations for changes, modifications, or corrective actions to improve data quality or efficiency in subsequent Passaic River data acquisition tasks.

A5 Problem Statement and Background

The USEPA, USACE, NJDOT-OMR, and the Trustees for Natural Resources [New Jersey Department of Environmental Protection (NJDEP), United States Fish and Wildlife Service (USFWS), and National Oceanic and Atmospheric Administration (NOAA)] have partnered to conduct a comprehensive study of the Lower Passaic River. The Lower Passaic River is the 17-mile tidally influenced stretch of the river from the Dundee Dam south to Newark Bay. The Lower Passaic River Restoration Project is an integrated, joint effort among state and federal agencies that will take a comprehensive look at the problems within the Lower Passaic River Basin and identify remediation and restoration options to address those problems. This multi-year study will provide opportunities for input from the public at all phases of development.

The Passaic River is the principle river (along with the Hackensack river) draining a 935 square mile watershed located in northern New Jersey and southern New York (see Figure A-2). All or portions of 117 municipalities in eight New Jersey counties, and 15 municipalities in two New York counties are located within the Hackensack-Passaic watershed (see Figure A-3). Due to historical contaminant releases, the Lower Passaic River sediments are contaminated with dioxins, PCBs, PAHs, metals, pesticides and other contaminants. As a result of the presence of these contaminants in biota, NJDEP has instituted a ‘do not eat’ advisory/prohibition for both fish and shellfish that inhabit the Passaic River (NJDEP and NJDHSS, 2004).

The Harrison Reach of the Lower Passaic River was selected as the location for the Environmental Dredging Pilot Study (see Figure A-4). The Harrison Reach extends approximately two miles from the NJ Turnpike Bridge to the Jackson Street Bridge that connects Harrison with Newark. The Pilot Study Survey Area is an approximately 1000-ft long stretch of the river within the Harrison Reach and is bordered to the north by the City of Kearny in Hudson County and to the south by the City of Newark in Essex County.

In 1994, Occidental Chemical Company (OCC) entered into an Administrative Order on Consent with USEPA. Chemical Land Holdings (CLH), on behalf of OCC, designed and executed a RI/FS work plan, which addressed the contaminated sediments of the Lower Passaic River in the vicinity of the former OCC facility in Newark, New Jersey. The

RI/FS primarily focused on the six mile reach of river extending upstream from the abandoned Conrail Railroad Bridge; that area has been designated the Passaic River Study Area (PRSA).

The contaminated sediments underlying the Lower Passaic River are of concern to various federal and state regulatory agencies because they can induce a number of negative consequences in the following areas:

- ecological health effects;
- human health effects; and
- economic impacts on navigational dredging and disposal costs in the NY/NJ Harbor.

As water quality, sediment quality and biological data from the RI/FS have become available, the scientific understanding of the Passaic River system has evolved and the potential importance of the inter-relationship of the 11 miles of the Lower Passaic River upstream of the PRSA and Hackensack River-Newark Bay system has become apparent. During the summer of 2001, USACE NY District completed a reconnaissance survey of the Lower Passaic River as part of their Hudson-Raritan Estuary Restoration Initiative. The USACE, USEPA Region 2 and NJDOT-OMR completed a Lower Passaic River Remediation and Ecosystem Restoration Project Management Plan (PMP) in April 2003. A National Memorandum of Understanding (MOU) was signed in July 2002 between USEPA and USACE. A Memorandum of Agreement among all of the agencies is pending.

NJDOT-OMR, USEPA, USACE, and the Trustees for Natural Resources recognizing the importance of the Lower Passaic River as an integral component of the Passaic - Hackensack - Newark Bay complex, have committed to better understand this system. The most significant potential benefit of addressing the environmental concerns facing the Lower Passaic River via a unified watershed approach is that the primary contaminants of concern can be addressed more effectively.

These Project Plans for the Environmental Dredging Pilot Study for the Lower Passaic River Restoration Project have been prepared by TAMS/ET and their subconsultant MPI for NJDOT-OMR as authorized under NJDOT Agreement No. 2001-NJMR02 Task Order #OMR-03-3.

The purpose of this task, which is described in this QAPP and the associated Work Plan, is to conduct an Environmental Dredging Demonstration and Sediment Decontamination Technology Demonstration. This work is part of the Lower Passaic River Restoration Feasibility Study, a joint effort of Federal and State Agencies to remediate and restore the Lower Passaic River Basin. The purpose of the overall Feasibility Study is to develop a comprehensive watershed-based plan for the remediation and restoration of the Lower Passaic River. During this pilot-scale demonstration project, approximately 5,000 cubic yards of contaminated sediment will be dredged from the Pilot Study Survey Area of the Passaic River. It is anticipated that this dredging will be performed in December 2005.

The objective of the dredging demonstration project is to study dredging productivity and sediment resuspension for the Lower Passaic River. Evaluating the feasibility of dredging requires the collection of data to determine the resuspension production rate, the resuspension release rate, and the resuspension export rate and to perform a mass balance. In addition, equipment performance, dredging production rates and turbidity levels will be evaluated. Dredging residuals will not be quantitatively monitored or evaluated as part of this pilot study. However, Sediment Profile Imagery (SPI) technology will be utilized to qualitatively evaluate the residual layer after dredging.

The Institute of Marine and Coastal Sciences at Rutgers University and the Water Resources Division of USGS, USEPA, and the USFWS will assist NJDOT-OMR in the sampling, monitoring, and other activities to be conducted during the Environmental Dredging Pilot Study.

The objective of the sediment decontamination technology demonstration project is to show that Passaic River sediments, contaminated with dioxins, PCBs, PAHs, metals, pesticides, herbicides, and other contaminants can be treated to meet applicable criteria for the appropriate beneficial use end product (e.g., cement, light weight aggregate, manufactured soil, glass, etc.). The decontamination project will collect data to perform a contaminant mass balance and determine the economic viability of the treatment process for commercial scale applications.

The Pilot Study consists of the following components:

1. Data Collection and Background – In order to establish baseline conditions and evaluate applicable technologies, several data collection and background activities have been completed. These activities include both general and site specific studies. Project Plans for Geophysical Surveys and Sediment Coring (TAMS/ET and MPI, June 2004a) and a Dredging Technology Review Report have been prepared by TAMS/ET and MPI (TAMS/ET and MPI, 2004b). A detailed characterization of the Pilot Study Survey Area has been presented in the Final Data Summary and Evaluation Report (DSER; TAMS/ET and MPI, 2005b).
2. Hydrodynamic Modeling – A hydrodynamic model (in support of the dredging pilot study) has been developed to provide an estimate of the amount of sediment leaving the study area and to aid in determining the placement of monitoring equipment. Modeling results are presented in the Environmental Dredging Pilot Study Hydrodynamic Modeling Report (ET, 2005).
3. Pilot Study Dredging Design – A design, including plans, specifications, and the NJDOT bid package (NJDOT, TAMS/ET and MPI, 2005), has been developed for the removal of approximately 5,000 cubic yards of contaminated river sediment from the Harrison Reach as part of the pilot dredging study. NJDOT used this design to procure the dredging contractor (Jay Cashman, Inc.).
4. Monitoring Plan – A monitoring plan has been prepared to outline procedures for the collection of data related to resuspension. The details of the monitoring plan are documented herein (i.e., the Work Plan, QAPP, and SWP). The analytical

methodologies selected will have sufficient sensitivity to meet these criteria for the analytes of interest in the water column and suspended sediments.

5. Decontamination – Two separate technologies, sediment washing and thermal treatment, will be employed to gather data regarding the effectiveness of decontamination. The decontamination technology vendors have prepared separate work plans that describe these efforts; these documents are currently under preparation and agency review.

As noted above, the work covered by this submission (Environmental Dredging Pilot Study project plans) is item 4. Items 1, 2, and 3 have been completed; and vendor submissions for item 5 are under review.

A6 Project/Task Description

A6.1 Description of Work to be Performed

The scope of work for this project, as defined in the associated Work Plan, includes:

- Monitor resuspended sediments (TSS) during dredging using a conductivity-temperature-depth (CTD) probe, Laser In-Situ Scattering and Transmissometry (LISST) probe, and optical back scatter (OBS) turbidity probe. Monitor water quality using the Trace Organic Platform Sampler (TOPS) apparatus. Deploy monitoring apparatus using fixed moorings and sampling boats. Evaluate sediment load by comparing TSS concentrations downstream to background TSS concentrations measured upstream. Hydrodynamic modeling previously completed (ET, 2005) was used in selection of the monitoring locations proposed in this plan.

- Concurrently with the work documented herein, develop a design to support environmental pilot dredging of contaminated sediment. As aspects of the design, prepare plans, specifications, and NJDOT bid package (TAMS/ET and MPI, 2005), which delineate the area to be dredged, required components of dredging equipment, and operational controls for the pilot study. Design and implement pilot dredging to mimic larger scale normal operations to the extent possible. Transport dredged sediments by barges/scows to sediment decontamination facility. Develop a sliding scale measurement and payment scheme, and implement by conducting pre- and post-dredging hydrographic surveys. Collect productivity data from contractor's daily reports.

- Interpret and evaluate the data and prepare a Summary Report that documents the work.

The sediment decontamination technology demonstration part of this project will be documented and performed in accordance with separate work plans developed by the specific decontamination technology vendors. The vendor plans are currently under agency review.

A6.2 Schedule

The proposed study schedule is provided as Table A-1.

A7 Quality Objectives and Criteria

A7.1 Data Quality Objectives

The design and implementation of the Environmental Dredging Pilot Study requires the collection of the following information about conditions in the Harrison Reach of the Passaic River:

- Water quality characteristics prior to, during, and after the five-day pilot dredging event (monitoring to be performed for an estimated maximum of eight days);
- Measuring the release (resuspension) of sediments during debris removal and dredging using both fixed moorings and shipboard surveys;
- Utilizing data obtained in this study as input for plans for subsequent investigations for other parts of the Passaic River; and
- Evaluating the data in the feasibility study to determine the monitoring and engineering control requirements for full-scale dredging.

The field and analytical work described in this QAPP is designed to generate sufficient data of adequate quality to achieve these DQOs.

A7.2 PARCC and Sensitivity - Definitions and Equations

Data quality and quantity are measured by comparison of resulting data with established acceptable limits for data precision, accuracy, representativeness, comparability and completeness (PARCC) and sensitivity. Data outside PARCC/sensitivity QA objectives will be evaluated according to Section B5 and the criteria contained in the specified analytical methods, to determine what, if any, aspects of the data can be defensibly used to meet the project objectives.

Data from previous investigations in the Harrison Reach of the Passaic River, available on the PREmis database, were reviewed in order to evaluate the appropriate criteria for the data to be generated, especially with regard to the sensitivity (detection and reporting limits) likely to be necessary. Only a very limited amount of water data was found. The draft final Pathways Analysis Report (PAR; Battelle, 2005) indicates that the ourPassaic.org database was searched but “All surface water data identified within the database were determined to be outside the boundaries of the study area.” A limited amount of data from the CARP program (2000 – 2003) were located; these data include some locations identified as being in the Passaic River and one sample location (Blanchard Street) near or in the Harrison Reach of the Passaic River. Unpublished data from these studies were reviewed qualitatively as an aid in assessing likely sensitivity requirements.

A7.2.1 Precision

Precision measures the reproducibility of data or measurements under specific conditions. Precision is a quantitative measure of the variability of a group of data compared to their average value. Duplicate precision is stated in terms of relative percent difference (RPD). Measurement of precision is dependent upon sampling technique and analytical method. Field duplicate and laboratory duplicate samples will be used to measure precision for project samples. Both sampling and analysis will be as consistent as possible. For a pair of measurements, RPD (or absolute difference; see Section B5.1) will be used, as presented below:

$$RPD(\%) = \frac{|D_1 - D_2|}{\left[\frac{(D_1 + D_2)}{2}\right]} \times 100$$

Where:

D_1 and D_2 = the two replicate values.

The upper limit for precision in non-aqueous matrix field duplicates is 100 percent RPD (in accordance with USEPA Region 2 data validation criteria for inorganics) for analytes present at five times the sample quantitation limit, and 50 percent RPD for aqueous duplicates. More detail on the implementation and evaluation of the precision criteria is presented in Section B5.1.

A7.2.2 Accuracy

Accuracy measures the bias in a measurement system that may result from sampling or analytical error. Sources of error that may contribute to poor accuracy are laboratory error, sampling inconsistency, field or laboratory contamination, sample handling, and matrix interference.

Equipment blanks, as well as matrix spike QC samples and Laboratory Control Samples (LCSs), will be used to measure accuracy for project samples. Accuracy is also assessed through the analysis of performance evaluation samples or standard reference materials samples, if used.

Accuracy is calculated using the equation below:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where:

%R = % recovery
SSR = spike sample result

SR = sample result
SA = amount of spike added to sample

Accuracy goals are method, analysis, and laboratory-specific. Accuracy is assessed quantitatively. Accuracy criteria for CLP methods are specified in the applicable CLP statement of work (SOW). For methods, analytes, or samples which do not have criteria specified in the CLP SOW, a default goal of 70 to 130 percent recovery is established unless the laboratory has developed its own recovery criteria.

A7.2.3 Representativeness

Representativeness expresses the degree to which sample data represent the characteristics of the media or matrix from which they are collected. Samples that are considered representative are properly collected to accurately characterize the nature and extent of contamination at a general sample location. Representativeness also requires that a sufficient number of data points (samples and analyses) be obtained so that the variability of the matrix being sampled can be ascertained to the extent required by the project objectives. Representativeness will be achieved by using standardized collection methods (e.g., sampling, handling, and preserving) and laboratory analytical methods. To the extent that the cited methods achieve project goals, methods for this dredging pilot study will be taken from those listed in Tables 4-1 (inorganics), 4-2 (organics), and 4-3 (wet chemistry) of the QAPP (MPI, 2005). Representativeness is assessed qualitatively.

A7.2.4 Comparability

Comparability expresses the confidence with which one data set can be compared with another data set from a different phase or from a different program. To achieve this, analytical methodologies used in this program are consistent with those used in previous investigations of the lower Passaic River, to the extent appropriate considering the objectives of the pilot dredging program. In some cases, newer or more sophisticated methods (e.g., PCB congeners by USEPA SOW CBC01.0) will be utilized in order to meet the sensitivity goals. Comparability is assessed qualitatively.

A7.2.5 Completeness

Completeness is defined as the percentage of data that is judged to be valid to achieve the objectives of the investigation compared to the total amount of data planned. Analytical completeness is the percentage of the usable data relative to the amount of data generated. Deficiencies in the data may be due to sampling techniques, poor accuracy, precision, or laboratory error. While the deficiencies may affect certain aspects of the data, usable data may still be extracted from applicable samples. An evaluation of completeness necessarily involves an evaluation of the impact of missing data on the ability of the project to achieve its goals. The goal for analytical completeness is 95 percent. The equation used for analytical completeness is presented below:

$$C(\%) = \frac{D \times 100}{P \times n}$$

Where:

C = Completeness

D = number of usable data points (includes both detections and non-detected results). Usable data are those with no qualifier; or with the U, J, or UJ qualifiers. (The 'D' flag, indicating a result from a dilution, is not considered a data quality qualifier; D-flagged data are also fully usable.)

P = number of analytical parameters per sample requested for analysis (e.g., seven for PCB aroclors by the CLP SOW [as there are seven different aroclors which are reported by that method])

n = number of samples requested for analysis

Overall completeness is calculated in a similar manner; except that the denominator is the number of data points planned. Overall completeness addresses both sampling and analytical completeness. The goal for overall completeness is 90 percent.

A7.2.6 Sensitivity

Sensitivity is defined as the ability to achieve the project-required reporting (quantitation) limits. Analytical methodologies are selected so that the target analytes will be detectable at the expected concentrations (based on review of limited available surface water data for the Harrison Reach of the Passaic River) or at the concentrations of concern (e.g., regulatory thresholds). For a given analytical method, quantitation limits may be adversely affected by matrix interferences, such as those caused by high concentrations of non-target analytes (e.g., sulfur or high molecular weight organics), or trace impurities in analytical reagents concentrated to detectable amounts in the analytical process. To control interferences, the laboratory will perform sample and extract cleanups (e.g., to remove sulfur) and laboratory, only pesticide grade or better solvents will be used, and method blanks must be demonstrated to be free of contamination prior to analysis.

In general, the methods selected for the project have been selected so that the target analytes will be reported at detectable concentrations in about 90 percent of the samples.

It is anticipated that the concentrations of contaminants in the media to be sampled and analyzed for this part of the project will be low. Therefore, although GC/MS methods allow the laboratory to screen samples prior to analysis, so that excessive levels of contaminants due not cause subsequent contamination problems in the laboratory (e.g., due to carryover, memory/ghost effects, and the like); such screening is unlikely to be necessary. In the unlikely event that screening is performed and indicates excessive contaminant concentrations, the initial analysis may be performed at a dilution (or use a smaller sample size), resulting in higher reporting limits for that sample. If a subsequent analysis is performed (due to one or more target analytes exceeding the calibration range), the laboratory will re-analyze the sample at an appropriate dilution factor, and report both results (diluted and undiluted) in the data package. Documentation of the screening performed to dilute the sample prior to the initial analysis will be provided by the laboratory and noted in the case narrative.

For non-routine organic analysis (dioxins and PCB congeners), recent USEPA CLP non-RAS methods may be used. For dioxins, CLP SOW DLM02.0 (Chlorinated Dibenzo-p-Dioxins [CDDs] and Chlorinated Dibenzofurans [CDFs], May 2005) may be utilized; the contract-required quantitation limit (CRQL) for that method is 1.0 ng/kg for non-aqueous methods, based on a 10 gram sample extracted and analyzed. This translates to detecting 0.010 ng of 2,3,7,8-TCDD in the sample, well below the project sensitivity goal of 5 ng/sample for the GFF and XAD resin media. Similarly, the CLP non-RAS SOW for PCB congeners, CBC01.0 (Chlorinated Biphenyl [CB] Congeners, May 2005) has CRQLs for individual congeners ranging from 0.5 to 50 pg/µL in the extract. Based on an injection volume of 1.0 or 2.0 µL, this translates to 1.0 to 100 pg/sample (based on the 2.0 µL injection), well below the project goal of 1 ng/congener/sample.

A8 Special Training/Certification

Field personnel will adhere to the procedures specified in the site-specific safe work plan (SWP), including having met the following requirements prior to the commencement of sampling:

A training course of at least 40 hours that meets the requirements specified in 29 CFR Part 1910.120(e) on safety and health at hazardous waste operations; and a refresher course of at least 8 hours that meets the requirements of 29 CFR Part 1910.120(e) on safety and health at hazardous waste operations within the last 12 months.

The Site Health and Safety Coordinator will be responsible for verifying that field personnel for each participating organization have current health and safety training prior to commencement of field sample collection activities. The health and safety training records will be maintained as discussed in the SWP. As noted in the SWP, personnel responsible for boat operations will have the required licenses and certifications.

No other specialized training is anticipated for this project. Field personnel performing sample collection and measurement activities will be properly trained in equipment use and procedures necessary for each task prior to entering the field. Each contractor/consultant will employ their internal processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training. Personnel involved in setting up and utilization of the TOPS apparatus must have prior hands-on experience in its use. Training courses or workshops on specific equipment, techniques or procedures will be documented. The requirements of this QAPP will be reviewed by management and field personnel of each participating organization to confirm that persons with appropriate credential and experience are assigned to the tasks to be performed. It will be the responsibility of the Field Team Leader to verify that field personnel understand and comply with the applicable QAPP requirements for their individual tasks.

Personnel who are responsible for performing laboratory analyses will be properly trained by the laboratory director or designee to conduct the various laboratory analyses described in this QAPP. The laboratory(ies) participating in this project will have training programs that are equivalent to those requirements in the National Environmental Laboratory Accreditation Conference (NELAC/NELAP) standards, Section 5.0 Quality Systems, or other accreditation acceptable to USACE. The laboratory(ies) will have sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned functions. Data verification and validation not performed by USEPA will be under the direction of the QA Program Manager who is experienced with the production, reporting, verification and validation of analytical data.

A9 Documentation and Records

This QAPP will be distributed to each of the agencies and contractors responsible in the collection, generation and interpretation of field and analytical data. The QA Program Manager will be responsible for verifying that necessary changes occur to keep the QAPP current with actual practices. The QA Program Manager will maintain a distribution list of QAPP recipient organizations or individuals so that revisions and updates can be distributed. The document control format used in this QAPP will identify the QAPP revision number and revision date. A QAPP revision history will be maintained that identifies each revision and a summary of the revision. This revision history will be incorporated into this section of the QAPP in any subsequent issues of the revised or updated QAPP.

- Revision 0, June 16, 2005. This is the initial document submitted to NJDOT-OMR, USEPA and USACE for review and comment.
- Revision 1, July 7, 2005. Internal review copy, not submitted for agency review.
- Revision 2, November 18, 2005. Final copy, addressing agency comments.

Analytical data for this project will be reported in both an Electronic Data Deliverable (EDD) and analytical data package. The EDD will be generated by the participating laboratories and will be used by the MPI (responsible for data management) to facilitate loading the analytical data into the project database.

Appropriate records will be maintained to provide adequate documentation of the entire data generation process, including field sampling and laboratory analysis. Field sampling records will include maintaining field logs and sample chain of custody documentation (see discussion in Section B of this QAPP).

The final evidence file will be the central repository for documents that constitute evidence relevant to sampling and analysis activities as described in this QAPP and the Work Plan. TAMS/ET and MPI and the various consultants/contractors are custodians of and will maintain the contents of the evidence files for the sediment sampling and analysis program, including all relevant records, correspondence, reports, logs, data, field records, pictures, subcontractor reports, analytical data, and data reviews.

The final evidence file will include, as applicable, the following:

- Field records;
- Field data and data deliverables;
- Photographs;
- Drawings;
- Laboratory data deliverables;
- Data validation reports;
- Field and laboratory audit reports, if any,
- Progress reports, QA reports; and
- Custody documentation.

B. DATA ACQUISITION

B1 Sampling Process Design

The Lower Passaic River Environmental Dredging Pilot Study consists of a single data acquisition task: surface water characterization (resuspension monitoring). The experimental design is provided below.

B1.1 Resuspension Monitoring Rationale/Plan

As dredging is one of the technologies that will be evaluated as part of the feasibility study for the Lower Passaic River site, part of the goal of the dredging pilot study is to estimate the amount of sediment resuspension and subsequent transport that might occur as a result of dredging operations in the Passaic River estuary. The rationale and approach for the monitoring plan presented herein is described in detail in sections 4.1 and 4.2 of the Work Plan (included in this submission) and is not repeated here.

B1.2 Description of Sampling Apparatus

To monitor TSS load and water quality, field equipment needs to be deployed in the near-field area (both upriver and downriver) to monitor water column stratification and stability; particle concentration and size distribution; and dissolved contaminant concentrations. The following discussion briefly describes each of the major instruments and tools to be used in the monitoring program.

The necessary data will be obtained from a combination of field instruments deployed at six mooring locations within the dredge pilot study area, field data collected by monitoring boats (along with some confirmation samples to correlate field data with laboratory TSS analysis), and by collection of samples for chemical (laboratory) analysis from sampling equipment deployed on other vessels. The apparatus for collection of each of these data types is described below.

B1.2.1 Field Instruments at Fixed Moorings

Several instruments will be deployed at a total of six mooring locations (three upstream and three downstream of the area to be dredged). These include conductivity-temperature-depth probes; LISST Probes; and optical backscatter (OBS, or turbidity) probes. The physical layout of the instruments is shown on Figures B-1 and B-2; and a schematic of the instrument deployment at each mooring is shown on Figure B-3. These instruments will be deployed prior to the initiation of any dredging-related activities (including debris removal) and will continue for at least one day after the completion of the pilot dredging program. Data from these instruments will be downloaded after the instruments are retrieved at the completion of the program.

B1.2.1.1 Conductivity –Temperature-Depth Probe

A conductivity-temperature-depth (CTD) probe is a self-contained probe and data logger that collects conductivity, temperature, and pressure measurements. Data are collected simultaneously and continuously as the CTD instrument is lowered through the water column. Typically, conductivity is measured with inductive coils; temperature is measured with a resistance thermometer; and pressure is measured with a strain gauge transducer. Salinity is then deduced from the conductivity value, and depth is deduced from the pressure reading. Together, these data can generate a density profile in the water column, which can provide information on water stratification and water column stability.

Two CTD probes will be deployed at each mooring (for a total of 12 instruments). The lower one at each mooring will be located just above the tripod (about 1 m above the river bottom) and the second will be tied to the tripod but will be suspended about 1 m from the river surface by a float. In addition, the "M" boat will be outfitted with a CDT probe and will acquire real-time data during dredging operations.

The SOP for CTD deployment and data acquisition (Procedure #PR 27) is provided in Appendix A (initially provided in Attachment 2 of the Draft FSP, Volume 1 [MPI, 2005b]).

B1.2.1.2 LISST Probe and Turbidity Meter

Both the LISST probe and the turbidity meter collect data on TSS. The LISST probe is a laser-based sediment sensing instrument (Sequoia Scientific, Bellevue, WA); the simplest LISST probe is a self-contained, data logger that measures *in situ* particle size and particle size distribution in the water column. The probe basically operates by shining a laser beam through the water column. The beam is then diffracted by the particles in the water column. The angle of diffraction and the intensity of the diffracted light are mathematically related to the particle size and the number of particles in water column. The user's manual for the LISST sampler is included in Appendix A. The LISST probe will be mounted near the surface of the water (about 1 m below the surface) to prevent instrument failure due to high loads at the river bottom, as the laser cannot penetrate suspended solids concentrations greater than about 100 mg/L.

A turbidity meter measures the clarity in the water column using optical properties (i.e., optical back-scatter, or OBS). A water sample with no suspended solids, or high clarity, will transmit light in a straight line. As the clarity of the sample decreases, or the mass of suspended solids increases, the amount of light scattering will increase. Turbidity is traditionally reported in nephelometric turbidity units (NTU), which represents the average volume scattering over a defined angular range. Together, data collected from the LISST probe and turbidity meter provide real-time information on the TSS in the water column. The turbidity meter along with the CTD probe form the main monitoring suite for this study. The SOP for acquisition of turbidity data (Procedure #PR 30) is provided in Appendix A.

B1.2.1.3 Acoustic Doppler Current Profiler

The Acoustic Doppler Current Profiler (ADCP) measures the speed and direction of ocean currents using the principle of “Doppler shift”. The ADCP exploits the Doppler effect by emitting a sequence of high frequency pulses of sound that scatter off of moving particles in the water. Depending on whether the particles are moving toward or away from the sound source, the frequency, or pitch, of the return signal bounced back to the ADCP is either higher or lower. Particles moving away from the instrument produce a lower frequency return and vice versa. Since the particles move at the same speed as the water that carries them, the frequency shift is proportional to the speed of the water, or current. The ADCP has four acoustic transducers that emit and receive acoustical pulses from four different directions. Current direction is computed by using trigonometric relations to convert the return signal from the four transducers to ‘earth’ coordinates (north-south, east-west and up-down) (NOAA, 2005).

One acoustic Doppler current profiler will be deployed at the base of each of the six moorings, mounted on the tripod about 1 m above the river bottom.

The SOP for acquisition of data using the ADCP (Procedure #PR 26) is provided in Appendix A (initially provided in Attachment 2 of the Draft FSP, Volume 1 [MPI, 2005b]).

B1.2.1.4 Trace Organics Platform Sampler

A Trace Organics Platform Sampler (TOPS) is used for collection of large-volume samples for measurement of low-concentration organic contaminants in the water column. The TOPS sampler was developed by NYSDEC personnel but has also been utilized by USGS and Stevens Institute of Technology personnel; USGS will have the responsibility for the implementation of the TOPS for this Environmental Dredging Pilot Study. Since the concentration of some hydrophobic compounds in the water column is low, laboratory extraction procedures involve large quantities of water in order to concentrate and to detect these compounds. To eliminate problems associated with the collection, handling and transport of a large volume (e.g., 50 liter) sample, TOPS operates by pumping water in the field through a non-ionic polymeric XAD resin absorbent cartridge. Hydrophobic organic compounds then partition from the dissolve phase to the polymer. Contaminants remain on the polymer until they are eluted at the analytical laboratory and concentrated with solvents. The TOPS is also equipped with a glass fiber filter (GFF) to collect particles for chemical analysis of suspended solids, and with a flow meter to measure the amount of water sampled. (The nominal GFF pore size [0.7 µm] is slightly larger than the nominal pore size [0.45 µm] used for filtering water samples for metals, so two are not identical.) For this project, the TOPS will be equipped with a splitter (bypassing the filters and XAD resin cartridge) for collection of samples for metals analysis. The general layout of the equipment in the TOPS vessel is shown on Figure B5, and a schematic of the TOPS apparatus is shown in Figure B6. The SOP for the operation of the TOPS apparatus is provided in Appendix C (USGS SOP #3).

B1.2.1.5 ISCO Sampler

A pair of ISCO samplers, provided by USGS, will also be utilized on each of the two vessels on which the TOPS apparatus is deployed (i.e., four ISCOs in all). One ISCO sampler will be used to collect samples for TSS analysis; and the other ISCO sampler will collect samples for organic carbon analysis. The ISCO samplers will be in operation concurrently with the TOPS sampler, so that the time periods over which the TSS and organic carbon samples are collected coincide with the time periods over which the water is passing through the TOPS. A separate, discrete sample pair (i.e., one TSS and one organic carbon [the sample for BR/Cl analysis will be taken from the sample used for organic carbon analysis]) will be collected from the ISCO sampler for each of the leg of the seven transects made by the TOPS vessel for each three-hour sampling event; a separate sample will be collected from the leg for which the sample is collected from the 1-m depth and another sample from the return leg during which the sample is collected from about 1-m above the river bottom. (This will result in 14 discrete TSS and organic carbon-Br/Cl samples collected for each 3-hour event; however, not all samples collected will be analyzed for organic carbon and Br/Cl.)

The ISCO samples will not be composite but submitted and analyzed as discrete samples. The ISCO sampler utilizes special bottles (wedge-shaped plastic, about 1-L capacity, designed to fit the configuration of the ISCO sampler); these bottles will be used as the sample bottles and will be shipped to the USGS laboratory in Trenton. The organic carbon samples will be filtered by USGS personnel, generating the filters for particulate organic carbon analysis and the filtered water for dissolved organic carbon analysis and Br/Cl analysis. The USGS laboratory will then package the filters for POC analysis and ship them to the DESA laboratory in Edison NJ. Similarly, the USGS laboratory will ship the filtered water to DESA for DOC and Br/Cl analysis. Separate bottles will be generated for DOC and Br/Cl (separate 250-mL bottles); the DOC samples will be preserved with H_2SO_4 after filtration.

The ISCO samplers do sometimes have problems with acquiring sufficient water (although this problem is expected to be lessened by the fact that the water samples are being collected from relatively near-surface depths). The paired ISCO samplers on each TOPS vessel provide redundancy, as both the TSS and organic carbon analyses can be performed on a single sample if one of the ISCO samplers does not retrieve adequate volume. The SOP for the ISCO sampler is provided in Appendix C (USGS SOP #4).

B1.2.2 Vessel-Mounted Field Instruments

In addition to the data collected from the instruments at fixed moorings, additional data will be collected during the time that dredging is underway. Due to the different speeds necessary for collection of these data, two different boats will be utilized. These two boats are referred to as the “Monitoring” or “M” boat, and the “LISST” or “L” boat. The path or track of the M boat will be perpendicular to the plume (i.e., will run generally cross-current; while the path of the L boat will be primarily parallel to the flow, although the L boat will also make some cross-current traverses to monitor the edge of the plume. However, the depending on the tidal cycle (ebb or flood), these boats will operate either on different sides of the dredge – downstream of the direction in which the dredge plume is anticipated. The general approach for the tracks of the L and M boats are shown on Figures B-1 (ebb tide) and B-2 (flood tide). Data from the "M" and "L" boats will be available in real time.

Vessel-mounted instruments include the CTD, LISST, and ADCP. The principle and operation of these instruments is described above (section 1.2.1), and the deployment described below (section 1.3)..

B1.3 Placement of Equipment and Frequency of Sampling

The near-field plume of TSS will exist either upriver or downriver, following the tidal movement of water in the river. During ebb tide, the plume will stretch downriver, and the outgoing water currents will transport TSS downriver. When the tide changes to a flood tide, the plume will stretch upriver and the incoming water currents will transport TSS upriver. In between these two tidal extremes, or slack tide, the plume will exist as a cloud around the dredging equipment. Due to the likely estuarine conditions on the Passaic River (i.e., the occurrence of water column stratification due to salinity), the leading edge of the near-field plume will occur at an angle as the overlying water moves at a different velocity than the underlying water. Based on observations and hydrodynamic modeling, flow in the water column is expected to be unidirectional, somewhat simplifying the monitoring program layout.

Each monitoring sweep of the near-field plume is expected to take about three hours, with several sweeps conducted during a single study period. It is expected that near-field monitoring will be conducted over eight tidal cycles during the five-day dredging program; however, field conditions may require modifications to this plan. During the three-hour monitoring period, the following activities will occur in the near-field plume:

- A sampling boat equipped with a CTD probe, turbidity (optical back-scatter, or OBS) meter and ACDCP (referred to as the "M" boat) will sweep across the near-field plume in a zig-zag pattern (Figures B1 and B2). The boat should cross the plume approximately seven times. The M boat will run five days during dredging (capturing a single tide event on Monday and Friday; and all day Tuesday, Wednesday, and Thursday).
- Another sampling boat equipped with the LISST probe (referred to as the "L" boat) will run generally parallel to river flow through the center of the plume, but will also sweep across the plume in a zig-zag pattern (Figures B1 and B2). It is anticipated that this LISST boat will be traveling at a slower speed than the CTD/turbidity boat. The L boat will operate concurrently with the M boat (five days during dredging).
- Grab samples (whole water) for TSS analysis will be collected periodically from the "L" boat and "M" boat. These samples are specifically for calibration of the instruments on the L and M boats; i.e., the instrument readings will be correlated to the laboratory TSS determinations from these samples. It is estimated that

approximately 50 samples (25 from the M boat, and 25 from the L boat) will be collected for TSS analysis over the course of the entire pilot dredge program; these are in addition to the samples for TSS analysis collected from the TOPS boats.

- Two sampling boats equipped with the TOPS apparatus and other sampling equipment will be deployed (the TOPS boats). Each will conduct traverses across the river at half-hour intervals during each three-hour sampling event. Each will be deployed about 300 m from the dredging operations, on opposite sides (i.e., one 300 m upstream, and one 300 m downstream) as shown on Figures B1 and B2.
- Three anchored moorings will be stationed within the near-field plume to collect continuous water column data (Figures B1, B2, and B3) on either side of the dredging operation (i.e., a total of six moorings). These moorings will be equipped with turbidity meters, and CTD probes. One of the three moorings on each side of the dredging operation will be equipped with a single LISST probe and an ADCP instrument (acoustic Doppler current profiler, which measures the current in the entire water column). An ADCP, OBS, and CDT will be located at the bottom of the tripod, about 0.5 to 1 m off the bottom of the river. Attached to the tripod, but suspended near the surface of the water (about 1 m below the surface) by means of a float, will be an OBS, CDT, and LISST. The typical deployment of instruments on a single mooring is illustrated on Figure B4.
- A turbidity probe or a LISST probe will also be attached to the dredge boat to collect TSS data adjacent to the dredging equipment.

To compare the TSS load in the near-field plume with background loading, data will be collected on the opposite side of the dredging activities (Figures B1 and B2).

- One sampling boat equipped with the TOPS instrument will either be anchored or move back and forth along a transect located approximately 300 m from the dredging activities. The TOPS boat will collect samples for chemical analysis (organics, metals, and wet chemistry, as described in greater detail below). A single sample will be collected at each TOPS boat during each three-hour event for organic chemical contaminants (pesticides; PCB congeners; and CDDs/CDFs). Paired 'whole' and 'dissolved' water samples for metals (TAL ICP-MS metals (iron by OES) except mercury) analysis will be collected concurrently (one pair from each boat for each three-hour event). Only a whole water sample is collected for the sample to be analyzed for low-concentration mercury (by Method 1631E) to reduce the potential for introducing ambient contamination from handling of the sample and filtration equipment in the field. A limited number of additional samples will be collected for dissolved mercury analysis; these samples will be collected as whole water samples (unpreserved) and shipped within 24 hours of collection to the laboratory. These samples will be filtered in the laboratory for dissolved mercury analysis.
- Two ISCO samplers will be deployed on each TOPS vessel (see Section B1.2.1.5, above). The ISCO samplers will collect discrete samples (one for TSS and one for organic carbon analysis and Br/Cl) for each leg of each transects (one across ["outbound"], and one return) made by the TOPS vessel during continuously

throughout each day (not just during the three-hour chemical sampling events). Based on 10-hour days, with the first transect at 7:00 am and the last at 5:00 pm, there will be 21 transects by each boat, with two legs per transect, for a total of 84 samples form each of the two ISCO samplers.

- Three anchored moorings will be stationed within 300 meters of the dredging activities (Figures B1 and B2). These moorings will be equipped with turbidity meters, CTD probes, LISST probe, and an ADCP instrument (Figures B3 and B4), and they will collect upstream (background) data on TSS data and water column conditions. This array will be a mirror image of the downstream array of moorings. When the tide reverses direction, the roles of the two sets of three moorings will be reversed as the upstream set is now located within the plume and the downstream set will observe background conditions.

B1.3.1 Schedule and Sequence of Sampling Events

A conceptual schedule showing the types and quantity of environmental samples collected during each day of the program is summarized below. A practice/dry run day (in which all the boats will be in the water, but no samples will be collected) has been tentatively scheduled for Wednesday, November 30, 2005 (the practice day is not included in the day-by-day summary below). The sampling for chemical analysis (conducted from both of the TOPS boats for all days except the post-dredge day, during which only one TOPS boat will be used) will be conducted during 3-hour events (typically; some will be shorter) which consist of transects conducted for the three-hour period immediately to either high tide or low tide. Based on the tide charts and the daylight hours in Newark the week of December 5 (sunrise at about 7:05 am and sunset at about 4:30 pm), the following schedule for the collection of samples for chemical analysis from the TOPS boat has been developed. (Note that for all days of dredging, all sampling boats will be in the water and collecting instrument readings and TSS samples at 7:00 am, one-half to one hour prior to the start of dredging.)

Day 1, Pre-Dredge Day (Thursday, December 1) – The dredging contractor has not yet begun any operations. Monitoring will occur on this day to establish ‘background’ (pre-dredge) conditions. Moorings (and associated field instruments) will be deployed on or prior to this day. One TOPS sampling event (two T-boats) will be conducted to establish ‘background’ conditions. The TOPS boat will make one traverse (transect) back and forth across the river (perpendicular to river flow) every 30 minutes during each three-hour sampling event, so that a total of seven traverses will be made for each event. It is estimated that a traverse will take about 10 or 12 minutes (about five or six minutes each direction). A single sample for each event (integrated [composited] over the six or seven traverses) will be collected and analyzed for dissolved and total metals; mercury; pesticides; PCB congeners; and PCDDs/PDCFs. Discrete samples from each leg of each traverse (each leg being one-half the round trip back and forth across the river; each leg will represent either the 1-m below surface depth interval, or the approximately 1- to 1.5-m above the sediment interface depth interval) will also be collected for TOC/POC/DOC analysis (at the same frequency as those for TSS) and continuous composite samples (composited over the 5- or 6-minute estimated duration of each of the two legs comprising a single traverse, with traverses conducted at 30-minute intervals throughout the day. Note that it is not anticipated that the sampling will be conducted over the full 10-hour period on the pre-dredge day, so the actual number of TSS and OC/Br/Cl samples collected will be determined by the actual number of transects conducted (which will not be less than the seven needed to get a full 3-hour TOPS sample).

Day 2 – First day of sediment dredging (assumed to be Monday, December 5). This will likely be the startup/shakedown day for the dredging contractor. Based on the tide cycle and available daylight, two TOPS events will occur on this day. The first will be a pre-high tide event, consisting of six transects (beginning at 8:00 with the last one at 10:30) prior to high tide at 10:46. A pre-low tide TOPS event will also be conducted (terminating slightly early due to darkness; this event will consist of six transects from 3:30 pm through 5:00 pm (low tide is at 5:48). Samples collected on the first day of dredging will be analyzed for total and dissolved metals; pesticides, PCB congeners and PCDDs/PDCFs. A set of discrete samples (one from each leg of each traverse throughout the day, assumed to be 21 transects beginning at 7 am and ending at 5 pm) will be collected from each of the two TOPS boats for TSS, POC/DOC, and Br/Cl analysis. so 84 discrete samples for TSS analysis are generated for each 10-hour day (21 transects; two legs [samples] per transect; from each of two boats).

Days 3, Second day of dredging (assumed to be Tuesday, December 6). Two TOPS events will be conducted this day. The first is a typical three-hour sampling event will be conducted prior to high tide (11:49 am; i.e., sampling transects from 8:30 through 11:30 am). On this day (and this day only) an additional sample will be taken bracketing the peak flow period (i.e., the midpoint between high tide and low tide.) On this day, the midpoint between the 11:49 high tide and the next low tide (6:45 pm) is about 3:17 pm; bracketing this point results in a set of seven transects from 1:30 to 4:30 pm. One set of samples will be collected from each TOPS boat for all parameters for each of the two events TOPS events conducted this day. TSS and OC/Br/Cl samples will be collected from each leg of each transect from each TOPS boat.

Days 4 and 5 – Third and fourth days of dredging (assumed to be Wednesday and Thursday). A single three-hour sampling event will be conducted on each of these days; each one prior to high tide (12:50 pm, and 1:49 pm). One set of samples will be collected from each TOPS boat for all chemistry parameters.

Day 6 – Fifth (and final) day of dredging (assumed to be Friday, December 9). Two TOPS sample events will be conducted on this day. In order to obtain a second pre-low-tide event sample, the dredger will start at 7:30 on this day, in order that the TOPS sample boats can complete four transects (starting at 7:30) prior to low tide at 9:21 am. The second TOPS event on this day will be a full set of seven transects (from 11:30 through 2:30) covering the 3-hour period prior to high tide at 2:49 pm.

Day 7 – Post-dredging day (Monday, December 12). Final field data collection; moorings and instruments retrieved and data downloaded. One last TOPS vessel sampling event (from 9:00 through 12:00, prior to low tide at 12:10 pm), collect samples for TSS, metals, pesticides to verify that the system has returned to pre-dredge conditions. It is anticipated that only the seven transects for a single TOPS sample will be conducted on this day (so only 28 samples for TSS will be generated).

In the schedule above, a total of ten 3-hour TOPS events are identified, with two samples (one from each of the two boats) for all days except the post dredge day, for a total of 19 environmental samples.

B1.3.2 Establishing Sampling Locations

Horizontal position of the sampling locations (including moorings) will be obtained using global positioning methods (GPS) accurate to 1 meter or less horizontally. Each boat (each of the two TOPS vessels; L Boat; M Boat) have GPS capability for establishing its horizontal location. The TOPS boats will also obtain approximate information on the river bottom depth using commercial fish sounders (so that the weighted 'fish' with the intake water samples can be maintained at the designated depths above the river bottom). During the pre-dredge day or practice day, buoys will be placed at the 6-m depth at each end of the transects for the two TOPS boats. Each day, the TOPS boats will also position buoys at the maximum depth location during the first leg of the day; these temporary buoys will be removed at the end of each day (during the last transect).

In addition to establishing the locations of the vessels used for sampling and monitoring, a separate hydrographic survey will be conducted (by ASI) of the pilot dredge area in the Harrison Reach. Prior to initiation of the pilot dredging study, this survey will establish the pre-dredge elevations. Subsequent to the completion of pilot dredging operations, a post-dredge survey will be conducted to establish the final elevations.

The coordinates will be reported in horizontal datum NAD 83 and NJ State Plane feet and vertical datum NGVD 1929. The minimum curacy of the hydrographic survey will be as follows:

- Depth of water: ± 0.5 feet (0.3 m)
- Horizontal position: ± 3 ft (1 m), although the ASI equipment has the capability of 1 cm resolution

B1.4 Field Modification

Based on real-time data collected in the field, the monitoring approach may be modified in the field as necessary to best measure the plume. In particular, it may be necessary to modify the track, width, or number of traverses conducted by the monitoring and sampling vessels (L, M, and TOPS boats), based on field conditions and the capability of the vessels to maintain speed and position while accomplishing their intended data acquisition tasks. It is anticipated that the flow rate through the sampling apparatus will be adjusted in the field to compensate for the actual time of the traverses while still obtaining the planned sample volume. For the TOPS events for which less than seven traverses can be obtained, the flow rates will be increased proportionally.

B1.5 Investigation-Derived Waste Disposal

The sampling methods used for this program are not expected to generate any excess sample material; any excess river water can be returned to the river at the point of collection.

Other investigation-derived waste (IDW) materials (e.g., personnel protective gear, used sample tubing, etc.) will be rinsed so that it is visually clean (no significant sediment residue) and then disposed as ordinary (non-hazardous) trash.

It is not anticipated that any other IDW will be generated during the pilot dredging program.

B2 Sampling and Data Acquisition Methods

Several different types of sampling equipment and methods will be utilized for this program. These include field instruments for water quality data; grab sampling for resuspension data; ISCO samplers for TSS and organic carbon and Br/Cl samples; and the TOPS apparatus for trace organics and inorganics sampling. Each of these is described below.

B2.1 Field Data Acquisition

Field instruments will be utilized for collection of water quality data during this program. The data include temperature, conductivity, and depth data obtained using the CDT meter (described in section B1.2.1, above), and turbidity data from the LISST and turbidity meters (described in section B1.2.2, above). The field instrument data will be obtained from instruments affixed to the six moorings; and also from vessel-mounted instruments (i.e., on the L and M boats).

Samples collected by the TOPS apparatus require concurrent flow rate/total water volume data for the water passing through the sample media (GFF and XAD cartridges). The placement of these instruments and the timing and manner in which these data will be acquired were described in section B1.3 and is not repeated here.

B2.2 Sampling for Organics, Metals, and Wet Chemistry using the TOPS

The TOPS boat will be equipped with four separate sample intakes (i.e., tubes) attached to a weighted ‘fish’, so that the samples for all analyses will be collected from essentially the same water. One intake will be used for the samples for TSS analysis (ISCO #1), one for the samples for organic carbon and Br/Cl (ISCO #2), one for the sample for metals (TAL and Hg; dissolved and total), and the fourth for the organics (both dissolved and suspended). The sample intervals for all analyses will be concurrent; i.e., sample collection and termination will be at the same time for each type of analysis. In that way, the data all will characterize the same water.

In order to provide a reasonably (though imperfect) representation of river conditions with depth, the TOPS boat will deploy the sample intakes (on the weighted 'fish') at a depth of about 1 m below the river surface on the first part of each traverse, and will attempt to deploy the sample intakes at a depth of about 1 to 1.5 m above the river bottom on the return part of the traverse. It is important that the weighted fish not actually contact the river bottom, as this would stir up sediments and result in water samples containing suspended sediment not related to the pilot dredging operation. To aid in maintaining the appropriate depth, marker buoys will be set up along the anticipated path of the TOPS traverse, and there will be a practice day (in which the boats conduct the traverses but samples are not collected) prior to the first day on which samples are collected.

B2.2.1 Sampling for TSS and Organic Carbon Using ISCO Samplers

The samples for TSS and organic carbon will be collected using two ISCO samplers by pumping (using a dedicated peristaltic pump) at a measured flow rate directly into a special wedge-shaped approximately 1-liter bottle. (Each TOPS vessel will be equipped with two ISCO samplers – one for TSS, and one for organic carbon and Br/Cl.) The flow rate of the peristaltic pump will be adjusted so that the filling of the bottle coincides with the time it takes the TOPS boat to complete one leg of the transect (either back or forth across the river). For example, if it takes about ten minutes for the boat to complete a transect (five minutes for each 'leg'), the flow rate for ISCO sample collection will be set at about 100 mL/minute. Since it may be difficult to time these transects accurately and consistently, additional bottles or partial bottles will be available to collect any additional volume in the event that the transect takes longer than planned. For example, if the flow rate has been set at 100 mL/min but the transect actually takes 12 minutes to complete (generating a total volume of 1200 mL), the last 200 mL will be collected in a second bottle; the two bottles (one full and one partial) will be combined in the laboratory for a single (TSS and/or organic carbon and Br/Cl) analysis representing that transect. The laboratories will be provided with instructions for combining the bottles to generate the appropriate 'complete' sample for each transect.

For these analyses, each leg (direction) is treated as a discrete sample; so each transect generates two sets of samples. For each three hour event (typically consisting of seven transects), a total of 14 samples are generated. All the discrete samples will be analyzed for TSS. However, to keep the organic carbon and Br/Cl analyses to a reasonable number, only half the samples will be filtered (the samples collected on the hour; the samples collected on the half hour will be retained but not filtered or analyzed). The filtration will be conducted by USGS (Trenton). For organic carbon, all the filters generated will be submitted for POC analysis (by DESA); and half the filtered water samples will be submitted for DOC and Br/Cl analysis (the other half of the filtered water samples will be saved at the USGS facility for later analysis if needed).

B2.2.2 Sampling for Metals

The sample for metals will collected (through a separate intake line) in a similar manner as the TSS sample. The major difference is that the sample for metals will be split into four bottles through "Y" fittings. One end of the "Y" will send the water directly to a (preserved) 1-liter sample bottle (for the whole water total metals analysis), and the other end of will send the sample through a 45 micron filter to a separate preserved bottle (for the dissolved metals analysis); i.e., the sample is filtered prior to contact with the preservative. Since two 1-liter bottles are being collected for metals, the flow rate for the metals sample will be set at two times that used for the TSS analysis. These samples will be collected in 2.5-L bottles; however, these bottles do not require the specialty cleaning needed for the trace-level mercury analysis.

A separate 2.5-L specially-cleaned bottle (glass or fluoropolymer) and tubing (Masterflex C-flex) is required for the dissolved and total trace level mercury analyses (i.e., samples collected for analysis by USEPA Method 1631E); these items are provided by the analytical laboratory. Note that in accordance with the method, the samples for trace mercury will not be field filtered; they will be shipped overnight to the laboratory, and the sample designated for dissolved mercury will be filtered in the laboratory. Due to the large volume collected (in the 2.5 L bottle), all the necessary analyses (whole water and dissolved; and MS/MSD as needed) will be taken from a single bottle.

Unlike the TSS organic carbon-Br/Cl analyses, each metals sample will be a composite of the three-hour event; so each bottle for metals analysis will be comprised of water collected over each of the 14 (typically) legs of the transects conducted during that event.

B2.2.3 Sampling for Organics

Samples for organic parameters (pesticides, PCBs, and dioxins/furans) will be collected through the TOPS apparatus. Suspended matter samples (glass fiber filters) will be collected for all 17 to 19 field samples, consisting of two (one from each boat) from the pre-dredge day; four (two from each boat) from the first and last days of the dredging (i.e., Monday and Friday; one approaching high tide and the other approaching low tide); and a total of six (one from each boat on each day) on the three days in the middle of the pilot dredging operation (Tuesday, Wednesday, and Thursday). Dissolved samples phase (XAD resin cartridges) will also be collected on a subset, consisting of eight samples (two from the pre-dredge day and then one each from the second, third, and fourth day [Tuesday through Thursday] of dredging). (The dissolved phase [XAD] samples are separate from, i.e., not combined with, the suspended phase [GFF] samples.) A final (19^{th}) GFF sample is also planned for the post-dredge day, in order evaluate the extent to which the post-dredge conditions are similar to pre-dredge conditions.

Unlike the TSS sample, the samples for organics are high volume composites representing an approximately three-hour interval of the dredging cycle. Six or seven transects will be conducted for each of the dredging composites. With a target (minimum) volume of about 50 liters through the TOPS apparatus, the flow rate will be set at about 1 liter per minute (based on an estimated transect time of 10 minutes, and six or seven transects for a total sampling time of 60 to 70 minutes). These composite samples for organics represent the water sample composites submitted for metals analysis.

B2.3 Grab Sampling for Resuspension Analyses

In addition to the TSS samples collected from the TOPS boats, approximately 25 to 50 1-L grab samples will be collected from the L and M boats for TSS analyses. The data from these samples, which will be collected from as near the instrument array on the moorings as practical, will be utilized to correlate the instrument data to TSS determined analytically.

B2.4 Decontamination Procedures

Decontamination of field equipment and its subsequent use will be documented in a field notebook. If visual signs such as discoloration indicate that decontamination was inadequate, the equipment will be decontaminated again. If the situation persists, the equipment will be taken out of service. Decontaminated sampling equipment will be wrapped in aluminum foil or otherwise protected to maintain its cleanliness when not in use.

Field decontamination of apparatus used to collect samples for chemical analysis will consist of a detergent (alconox) wash followed by rinsing with potable (tap) water, an acetone rinse, and distilled deionized analyte free water rinse. After decontamination, stainless steel apparatus and utensils will be allowed to air dry. If the equipment will not be re-used immediately, it will be wrapped and stored in aluminum foil or otherwise protected to maintain its cleanliness prior to subsequent re-use. Solvents and acids will not be utilized for equipment decontamination.

The initial decontamination of the sample bottles, glass fiber filters, and XAD resin cartridges will be performed by the analytical laboratory (STL, Knoxville, TN). Their SOP (KNOX-ID-0017) for the decontamination of the GFF and XAD is included in Attachment B. Sample bottles will be cleaned (and certified clean) to the requirements of the USEPA CLP (OSWER directive 9240.0-05A, Specifications and Guidance for Contaminant-Free Sample Containers). Decontamination of specialty bottles for trace level mercury analysis (1631E) will performed by the laboratory contracted to perform the analyses and will be consistent with the cleanliness and decontamination requirements specified in the method.

B3 Sample Handling and Custody Requirements

B3.1 Field Activities Sample Custody

The primary objective of sample custody procedures is to create an accurate written record which can be used to trace the possession and handling of samples from the moment of their collection, through analysis, until their final disposition. Due to the low concentrations of contaminants being measured in some media, it is also necessary to track the preparation, decontamination, and shipment of empty sample containers/media (glass fiber filters; XAD resin cartridges; "ultra-clean" bottles for trace mercury analysis); membrane filters for suspended metals sample analysis [if used]). As noted above (section B2.4), the initial decontamination of the bottles and sample media (GFF and XAD) will be performed by STL. These items will be shipped under custody to USGS, who will be responsible for the assembly of the TOPS apparatus while maintaining the integrity and cleanliness of the bottles and sampling media and equipment. The USGS SOP covering this aspect of the work is provided in Attachment A.

A sample (or sample container) is under custody if the following conditions are met:

- It is in the Field Team Leader's (or designee's) possession,
- It is in the Field Team Leader's (or designee's) view, after being in the Field Team Leader's (or designee's) possession,
- It was in the Field Team Leader's (or designee's) possession and the Field Team Leader (or a designee's) locked it up, or
- It is placed in a designated secure area by the individual who is maintaining custody.

A field log (Figure B-2) will be used to document custody of the samples from the time of collection until the initiation (preparation) of the formal chain-of-custody forms (Figure B-3). Separate field logs will be needed for samples collected on the TOPS boat and any samples (e.g., water samples for TSS) collected from the other boat. (Combined traffic report/chain-of-custody [TR/COC] forms are used for samples submitted to the CLP laboratory; see B3.1.1, below.). Custody for samples will be maintained by the field personnel collecting the samples. The field personnel are responsible for documenting each sample transfer and maintaining custody of samples until they are shipped or delivered by courier to the laboratory or archived or disposed.

The necessary sample containers will be shipped by a commercial delivery service (e.g.., FedEx) or delivered by laboratory courier to TAMS/ET and MPI personnel (the specific arrangements will be established after the laboratories have been selected). If sample containers are not provided by the analytical laboratories (as will be the case for CLP and DESA laboratories), sample containers that have been decontaminated in accordance with EPA specification (USEPA, 1980) may be purchased from commercial vendors. Certificates of analysis documenting the bottle cleanliness will be provided by the vendor and retained as part of project records. Sample container and preservation requirements are presented in Table B-2. The laboratory(ies) or bottle vendor will deliver containers at a frequency and location determined by the project team. Sample containers needed for a specific sampling task will then be relinquished to the sampling team after verifying the integrity of the containers and confirming that the proper bottles have been assigned for the task to be conducted.

Separate documentation will be required for the custody and decontamination of the critical parts of the TOPS apparatus, including the glass fiber filters and the XAD resin cartridges.

After a given sample has been prepared, a self-adhesive, waterproof label will be affixed to each container. At a minimum, the sample label will contain:

- Field sample identification number (in accordance with USEPA/CLP protocol, the site name is not to appear on any documentation sent to CLP laboratories),
- Date and time collected,
- Custodian's initials, and
- Analyses required

Immediately after sample preparation and labeling, each sample container designated for analysis will be sealed into a plastic bag and placed into an insulated cooler with "wet ice" or icepacks (for samples requiring temperature preservation) and appropriate packing materials for shipment to the laboratory.

A field chain-of-custody record (combined traffic report/chain of custody for CLP laboratory shipments) will accompany the samples to their destination. These record forms will be sealed in a plastic bag to protect them against moisture. The temperature of a temperature bottle blank will be monitored to verify that samples requiring temperature preservation are within 4° ± 2° C, as required, prior to leaving the site. Temperature blanks consist of bottles filled with distilled or tap water. The shipping coolers will then be sealed utilizing custody seals that will be initialed by the field personnel. Sample coolers will be delivered to the analytical laboratory by either direct courier or overnight delivery courier (e.g., FedEx) at the end of each day's sample processing activities.

B3.1.1 Shipping Documentation – CLP (including non-RAS CLP) Laboratories

It is anticipated that samples may be shipped to a single (non-RAS) CLP laboratory for extraction of the GFF and XAD resin samples for organics and analysis non-routine high resolution GC/MS (PCDDs/PCDFs and PCB congeners) organics and pesticides.

Required documentation includes the following:

- Custody seals – one on each bottle. Provided by the sampling organization. Signed (or initialed) and dated by sampler.
- Labels – one on each bottle. Provided by the sampling organization, generated by EPA Forms II Lite software. Includes the CLP Sample Number and the SMO-assigned Case Number (along with other information as described previously). (Note that the bottles and inner bag for trace mercury are not handled; labels and custody seals go on the outer bag.)
- Traffic Report/Chain of custody (TR/COC). One per cooler. Generated by Forms II Lite software.
- Custody Seals – two; on outside of cooler. Provided by sampling organization.

Note that USEPA Region 2 does not require the use of sample tags; and therefore sample tags will not be used. Also, CLP protocol specifies that the site name not appear on any of the documentation provided to the CLP laboratories.

Sampling personnel will report shipments to CLP laboratories to the EPA-designated RSCC/SMO contact by 8:00 a.m. of the day after the samples are shipped; and the TR/COC forms are exported electronically within five days.

B3.1.2 Shipping Documentation – USEPA Region 2 DESA Laboratory

Shipping documentation will be the same as for the CLP laboratories; except that RSCC/SMO reporting is not required. Although Forms II Lite is not required for DESA, it is anticipated that it will be utilized for convenience.

B3.1.3 Shipping Documentation – Commercial Laboratory

For samples shipped to the commercial (trace mercury) laboratory, the documentation is as follows:

- Labels – one on the outer bag containing each bottle. Provided by the analytical laboratory. (This is to minimize handling of the sample bottle; and to enable proper log-in at the laboratory, as log-in personnel are not allowed to open the plastic bags containing the trace mercury sample bottle.)
- Chain of custody form. One per cooler/shipping container. Provided by the TAMS/ET or the analytical laboratory.
- Custody Seals – two per cooler/shipping container; on outside of cooler. Provided by laboratory or sampling organization.

An example of a COC form which may be used for shipments to commercial laboratories is provided as Figure B-7.

B3.1.4 Shipping Documentation – USGS Laboratories

As of this writing (December 2005), no specific analyses have been assigned to USGS. However, the USGS laboratory in Trenton, NJ will be used to filter the water samples for POC and DOC analysis; the USGS laboratory will then ship the package and ship the filters and filtered water to the DESA laboratory for analysis. The USGS laboratories may be also be utilized as a backup for the TSS and organic carbon analyses. For samples shipped to USGS laboratories for organic carbon or TSS analyses, the documentation requirements are the same as for commercial laboratories as specified above (Section B3.1.3). Example paperwork and forms which may be utilized for samples shipped to USGS laboratories are provided in Appendix C (USGS SOP #2). The USGS laboratory will also retain extra samples from ISCO #2 (i.e., the samples collected on the half-hour for organic carbon and Br/Cl analyses; and the filtered water from samples not sent to DESA for dissolved organic carbon analysis).

B3.2 Laboratory Receipt and Custody

Once samples are received at the laboratory, the field chain-of-custody form (an example of which is provided as Figure B-7; TR/COC used for CLP shipments) is completed and signed by the individual Laboratory Sample Custodian. The Laboratory Sample Custodian will check the sample bottle labels against the corresponding information listed on the field chain-of-custody records and note any discrepancies. Additionally, the laboratory sample receipt personnel will note any damaged or missing sample containers. This information will be recorded on the field chain-of-custody record or in a separate logbook. The temperature of the temperature bottle blank included in each cooler of samples requiring temperature preservation will also be recorded at the time of sample receipt by the laboratory personnel. This temperature will also be measured on the field chain-of-custody record or in a separate logbook. Any discrepancies in sample identifications, sample analysis information, any indication that samples are missing upon receipt at the laboratory, or any indication that samples not received at the correct pH or temperature (4° ± 2° C) will be communicated to the QA Program Manager and Field Team Leader within 24 hours of sample receipt so that appropriate corrective action can be determined and implemented. For samples sent to CLP laboratories, the SOW requires that the laboratory contact the EPA SMO if the shipment temperature exceeds 10° C. Specific actions (e.g., decisions as to whether or not to analyze the sample) for samples outside the temperature criteria will be determined on a case-by-case basis by the QA program manager in consultation with NJDOT-OMR and USEPA Region 2 QA staff, as appropriate.

After the sample receipt information is checked and recorded, sample analysis information will be entered into the individual Laboratory Information Management System (LIMS) (or equivalent). Each sample will be provided a unique laboratory identification number and the analysis tests requested on the chain-of-custody records entered into the LIMS. After the required information has been entered into the LIMS, the Laboratory Sample Custodian will initiate an internal laboratory chain-of-custody. The internal chain-of-custody will document the transfer of samples from the storage location to the analyst for analysis and subsequently through final disposition at the laboratory. At a minimum, the internal chain-of-custody will include client identification, laboratory sample number, sample matrix, signatures for relinquishing and receiving samples or samples, and reasons for the change in custody.

It is currently planned that samples for the various analyses to be performed will be submitted in the containers as shown on Table B-2. However, this scheme is subject to change based on the number of different laboratories that will be used; the parameters analyzed by each laboratory; and the preferences of each laboratory performing the analyses.

All completed field and laboratory chain-of-custody records will be provided in the laboratory analysis data package as part of the required deliverable report.

Samples will be stored in secure, limited access areas in an environment that maintains any required temperature preservation noted in Table B-2. Samples for most analyses are required to be refrigerated at a temperature of 4° ± 2° C. (Analytical methodologies utilized do not specifically address the XAD resin and GFF sample matrices; for the purpose of this QAPP the requirements applicable to soil samples will be applied.) The temperature of the refrigerators or freezers used to store samples will be monitored by the project laboratories according to their internal standard operating procedures. Disposal of unused raw sample volumes, sample extracts, etc., will be in accordance with each laboratory's waste management procedures.

Samples for organic carbon and Br/Cl analysis will be processed at the USGS laboratory in Trenton. For these samples, a field-generated COC will accompany the samples from the field to the laboratory in Trenton. The USGS laboratory will sign for ('received by') the samples. At that point, the custody and processing of the samples will be documented by the USGS lab's internal custody documentation, and new custody documents will be generated by USGS to accompany the samples from USGS (Trenton) to DESA (Edison) for analysis.

B3.3 Extract and Sample Archive Procedures

Sample extracts will be held (frozen at ≤-10°C) at the laboratory until the final data reports have been validated and approved.

Archived extracts will be stored frozen in a clean and controlled manner at a temperature of ≤-10°C. Daily temperature measurements will be taken and recorded at the storage location using a NIST-calibrated thermometer to document proper temperature preservation. A label identical to that placed on the original sample will be used to identify the archive sample.

It is not planned to submit any water samples to be archived as part of the Environmental Dredging Pilot Study, although some extra sample volume (or extra bottles not slated for analysis) will be retained.

As noted above, extra sample volume from ISCO#2 and filtered water generated at the USGS laboratory will be retained (under refrigeration) temporarily at the USGS facility in Trenton. There are no plans to analyze these samples; however, they will be retained in the event of analytical problems or data anomalies which could be addressed by analysis of these retained samples. If there are no analytical problems or data anomalies with the samples analyzed for TSS, POC, DOC, and Br/Cl, then the material which had retained by USGS will be disposed.

B4 Analytical Procedures

The Passaic River Sampling and Analysis Program will involve the analysis of water samples for chemical constituents and water quality characterization. The justification and rationale for the selected analyses are presented in Section A7. The specific methods to be used are shown on Tables B-1 and B-2.

Several laboratories may provide analytical services for the pilot dredging program (see also table B-2). Laboratories which are currently projected to conduct the analyses for the pilot dredge program are listed below, along with the services which they may provide.

USEPA Region 2 Division of Environmental Science and Assessment (DESA) Laboratory, Edison NJ will perform organic carbon (filters for POC [DESA SOP C-88] and water for DOC [DESA SOP C-83]), TSS (DESA SOP C-33, based on EPA method 160.2), Br/Cl (EPA method 300.0) and dissolved and total metals (except mercury). Metals will be analyzed by ICP-MS; except for iron, which will be analyzed by ICP-OES.

USEPA Contract Laboratory Program laboratories may perform non-RAS organics analyses of the XAD resin and GFF media, including pesticides (CLP SOW SOM 01.1), CDD/CDFs (DLM02.0), and PCB congeners (CBC 01.0) analysis.

US Geological Service laboratories will perform the filtration of samples (and generate the filters and water) for TOC (POC and DOC) analysis, are backup providers who may perform TSS and organic carbon analyses.

A commercial laboratory (contracted by TAMS/ET under NJDOT-OMR contract) will be utilized as needed to perform specialized analyses beyond the scope or capacity of either DESA or CLP laboratories. These analyses will likely be limited to the low level mercury analysis of water samples (by EPA 1631E).

The chemical analyses to be performed collectively for water samples include PCBs (as PCB congeners); pesticides; metals (lead and mercury); CDDs/CDFs, and total suspended solids. “Water samples” as used here also includes the particulate phase retained on glass fiber filters or filters used for suspended matter analysis, and XAD resin cartridges through which large volume water samples have been passed.

The samples to be analyzed for pesticides, PCB congeners, and PCDDs/CDFs will be generated from high volume water samples collected from the TOPS apparatus. Each sample will be comprised of either a pair of glass fiber filters (the two together considered to represent the suspended matter phase), or a pair of XAD resin cartridges (installed in series; the combined mass on the two cartridges considered to represent the dissolved phase). Each sample (comprised of two GFF filters, or two XAD resin cartridges) will undergo a single extraction for all subsequent analysis for all three organic analytical fractions.

The procedures that will be used to for the analysis of the samples are summarized below. The analyses are grouped by general type: organics (section B4.1), inorganics/metals (section B4.2), and wet chemistry (section B4.3).

B4.1 Organic Analysis Procedures

Due to the nature of the samples (both the GFF and XAD resin), a single extraction will be performed for all three organic fractions (PCB congeners, dioxins/furans, and pesticides). A two-stage extraction will be performed by the analytical laboratory. The first stage is a hexane/acetone extraction (for pesticides, PCBs, and some dioxins), followed by a toluene extraction (for the remaining dioxins/furans). The first stage extract is used 'as is' for the PCB congener and pesticide analysis. An aliquot of the first stage extract is combined with the second stage extract for the CDD/CDF analysis. An example SOP for this extraction and analysis is provided in Attachment A.

As it is currently planned that these analyses will be performed by a CLP non-RAS laboratory, the laboratory performing the analysis will provide its own SOP for the extraction to USEPA for review prior to initiation of sample analysis.

B4.1.1 PCB Congeners

The USEPA Analytical Method for Chlorinated Biphenyl Congeners by EPA Method CLP SOW CBC 01.0 (May, 2005) will be used for PCB congener analysis. All 209 congeners will be reported; although due to coelutions, only about 160 discrete data points are reported..

B4.1.2 Polychlorinated Dibenzo-p-Dioxins/Polychlorinated Dibenzofurans

PCDD/PCDF analysis will be by CLP SOW DLM02.0 (May, 2005) for Analysis of Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs). All 17 2,3,7,8-substituted PCDDs/PDCFs will be reported; however, samples from the pilot dredging area are expected to be dominated by 2,3,7,8-TCDD.

B4.1.3 Pesticides

The extraction and analytical procedure for pesticides will be by CLP SOM01.0, Exhibit D, Analytical Methods for Pesticides (preferred); however, in the event that the laboratory performing the congener and CDD/CDF analysis cannot perform this method, CLP OLM04.3, Exhibit D, Analytical Methods for Pesticides/Aroclors for Pesticides analysis will be allowed. All target analytes will be reported; however, key analytes are 4,4'-DDT, DDE, and DDD; as well as chlordane. Based on initial conversations with a commercial laboratory and preliminary calculations of the expected minimum DDT loading on the GFF (suspended matter) samples, SW-846 method 8081(modified) will be sufficiently sensitive for this analysis. (The modification consists of concentration of the extract to 1 mL, rather than 10 mL. Alternatively, the laboratory may be asked to report non-detect values to the MDL, rather than the CRQL.)

B4.2 Inorganics/Metals Analysis

Metals to be analyzed for the pilot dredging program are TAL metals amenable to ICP-MS analysis (including aluminum and iron; iron will be analyzed by OES) and trace-level mercury.

B4.2.1 Lead and Other TAL Metals (Except Mercury)

Analysis for lead and most other TAL metals will be by ICP-MS (e.g., CLP SOW 05.3) for water. The USEPA Region 2 DESA laboratory is scheduled to perform these analyses; the DESA reporting limit for the TAL metals is 0.5 µg/L except for aluminum (60 µg/L) and iron (analyzed by ICP-OES, with a reporting limit of 100 µg/L).

B4.2.2 Mercury

The reporting limits specified in the CLP SOW ILM05.3 (200 ng/L) are much higher than the target dissolved phase concentration reporting limit (about 0.5 to 1.0 ng/L). As the desired reporting limit for mercury cannot be achieved using the CLP method, an alternate method, EPA Method 1631E, which has a reporting limit of 0.5 ng/L, will be utilized. It is anticipated that the low-level mercury analyses will be performed by a commercial laboratory (tentatively, STL, North Canton, OH).

B4.3 Wet Chemistry Analyses

Wet chemistry analyses performed for the pilot dredging program include organic carbon (particulate and dissolved [water]) and total suspended solids.

B4.3.1 Organic Carbon (Total, Dissolved, Particulate)

Total organic carbon (on particulate matter [filters]) will USEPA Region 2 DESA SOP C-88. Organic carbon analysis on water samples (dissolved organic carbon on filtered water samples) will be by DESA SOP C-83.

B4.3.2 Total Suspended Solids

Total suspended solids analysis will be by DESA SOP C-33 (based on USEPA MCAWW method 160.2). Ideally, the entire sample will be utilized for the analysis. However, if less than the full sample is used, the sample will be well-mixed (e.g., shaken vigorously) prior to removing an aliquot for analysis.

B5 Quality Control Requirements

Data Quality Objectives (DQOs) are quantitative and qualitative statements specifying the quality of the environmental data required to support the decision-making process. The intended use of data, analytical measurements and the availability of resources are an integral part in development of the DQOs. DQOs define the total uncertainty in the data that is acceptable for each specific activity during the sampling events. This uncertainty includes both sampling error and analytical instrument error. Ideally, the prospect of zero uncertainty is the intent; however, the variables associated with the collection process (field and laboratory) inherently contribute to the uncertainty of the data. The overall quality assurance objective is to keep the total uncertainty within an acceptable range that will not hinder the intended use of the data. In order to achieve this objective, specific data quality requirements such as detection limits, criteria for accuracy and precision, sample representativeness, data comparability, and data completeness will be specified. The objectives and requirements for this project have been established to provide a high degree of confidence in the data obtained.

B5.1 Field QA/QC Samples

QA/QC samples will be collected in the field to allow evaluation of data quality. Field QA/QC samples include equipment blanks, duplicate samples, matrix spike samples and matrix spike duplicate samples. The types and frequency of QA/QC samples to be collected for each parameter are described below, and illustrated on Table B-3. QA/QC samples prepared in the laboratory include method blanks, laboratory control spikes, and temperature blanks. Field quality control checks used during this investigation are discussed below.

B5.1.1 Equipment Blanks

The purpose of analyzing equipment blanks (rinsate blanks) is to demonstrate that sampling procedures do not result in contamination of the environmental samples and to evaluate the effectiveness of the decontamination of field equipment performed by field personnel. In general, equipment blanks will be prepared by processing a sample of laboratory grade analyte-free water in the same manner as environmental samples including tubing and pumps and placing in containers.

Equipment/Rinsate Blanks – TOPS Apparatus (Organics)
For the TOPS apparatus, there is a differentiation between field and rinsate blanks (i.e., the terms are not interchangeable). A rinsate blank will be generated for all organic parameters at the onset of field activities, or during the program, if possible (this would monitor for residual material not cleared from the system between samples) by passing clean (laboratory-provided analyte-free water) through the apparatus (the GFF and XAD resin cartridges) and submitting them to the laboratory. An equipment blank (field blank) will also be submitted; this will consist of a set of GFF filters or a pair of XAD resin cartridges (the same configuration as a field sample) which are handled similarly to an environmental sample, except that no river water is ever passed though them.

Equipment/Rinsate Blanks – Metals
Rinsate blank for metals analysis will be generated by passing clean (laboratory-provided analyte-free water) through the apparatus. The blank for total metals (whole water) will be generated by passing the water through the pump and tubing into the sample bottle.

For the dissolved (filtered) metals analysis, the blank water will also be passed through the filtration apparatus.

For low-level mercury analysis (method 1631E), a 'field blank' (which functions also as a 'trip blank', as its function is to "demonstrate that samples have not been contaminated by the sample collection or transport process." Field blanks (shipped with each set of samples) will be submitted at the frequency shown on Table B-2. A 'rinsate blank' will be collected at the outset of the program, utilizing mercury-free water passed through the pumps and tubing used for the mercury sample collection. It is anticipated that a second rinsate blank for the Hg analysis will be generated sometime near the middle of the dredging operations week.

Equipment/Rinsate Blanks – Wet Chemistry parameters
No equipment or rinsate blanks will be collected for the wet chemistry parameters, TSS and organic carbon.

Evaluation of field blank data
Field blank contamination will be addressed during data quality review. Data will not be "corrected" for blank contamination. Actions (data qualification) for field blank contamination will be similar to that discussed under method blanks, below (Section B5.2).

B5.1.2 Field Duplicates

The purpose of analyzing field duplicates is to demonstrate the precision of sampling and analytical processes. Field duplicates will be submitted at the rate of five percent of the total number of environmental samples (i.e., one duplicate for each group of 20 [or fewer] field samples). For water samples, the duplicates will not be homogenized but will be paired samples collected at the same time and location (technically, co-located samples rather than replicates). Meaningful analysis of a duplicate sample requires that detectable concentrations of the analytes are present in samples. The relative percent difference (RPD) of the two measurements on the sample is calculated by the following equation:

$$RPD(\%) = \frac{|D_1 - D_2|}{\left[\frac{(D_1 + D_2)}{2}\right]} \times 100$$

Where: D_1 = the measured value for the initial field sample

D_2 = the measured value for the field duplicate sample

Note: Precision in not calculable if the analyte is not detected in one or both analyses. In this case, precision is assessed qualitatively, not quantitatively. If the detected concentration in one or both analyses is less than five times the sample-specific reporting limit, precision is assessed based on the absolute difference (D) between the two results, not the RPD.

Field Duplicates - Organics
The TOPS apparatus is not amenable to collection of a field duplicate with a single setup; therefore, no true field duplicates will be collected. However, the samples collected from the two TOPS boats on the first day of sampling – before any dredging operations begin – should be similar if not identical, as the sample collected on both boats should be representative of ambient water conditions in the Harrison Reach at the time of collection.

Field Duplicates – Metals
Field duplicates for metals analysis will be generated by splitting the sample (by use of a "Y" connector) into two pairs of sample bottles (one directly into a pair of bottles for total metals analysis, and a second pair through (separate) filters into the sample bottles for the dissolved (filtered) metals analysis. Separate duplicates will be collected for mercury. The bottle labeled 'spare' on Figure B-6 will be used to collect the field duplicates.

Field Duplicates – Wet Chemistry parameters
Field duplicates will be collected for the wet chemistry parameters, TSS and organic carbon. TSS duplicates will be from the second ISCO sampler, using samples which were collected for TOC analysis but not submitted. TOC duplicates will be generated by the USGS laboratory (by submitting two filtered samples or two sets of filters generated from the same field sample). Br/Cl duplicates will be generated by splitting extra volume from the TOC sample.

Evaluation of Field Duplicate Data

If the RPD (or D) of field duplicate results is greater than the QC acceptance criteria, the environmental results for the field duplicate pair will be qualified as estimated. The USEPA Region 2 field duplicate precision criteria for metals will be utilized for all analytes (i.e., RPD of < 100 percent for non-aqueous samples; < 50 percent for aqueous samples; or D less than two times the reporting limit, as applicable).

B5.1.3 Matrix Spikes and Matrix Spike Duplicates

The purpose of analyzing matrix spike (MS) and matrix spike duplicate (MSD) samples is to assess analytical accuracy and recovery of analytes of interest in a particular sample matrix. A laboratory duplicates (LD) is typically substituted for MSD for inorganic and wet chemistry analyses. MS and MSD samples are not required for TSS.

MS/MSD/LD will be analyzed at the rate of one pair per sample batch (up to 20 samples) for organic chemical analyses, except for Chlorinated Dibenzo-p-Dioxins/ Chlorinated Dibenzofurans. MS and MSD is not required based on CLP SOW DLM02.0 (an LCS is required for each SDG) or 1613B (the method has alternate protocols for spiking and quality control sample analysis).

Each MS/MSD will consist of an aliquot of laboratory-fortified environmental sample. Preferably a sample of low contaminant concentration should be used so that the spike level is of sufficient concentration over the contaminant concentration of the chosen sample. Spike concentrations are given in the analytical SOPs. The MS and MSD are extracted and analyzed following procedures used for actual sample analysis. The percent recovery of the MS/MSD is calculated by the following equation:

$$\%REC = (A-B)/T \times 100\%$$

Where: A = concentration of analyte in the spike sample aliquot
B = background concentration of compound or analyte in the unspiked sample aliquot
T = known true value of the spike concentration

Matrix spike recovery information is used to assess the long-term accuracy of a method. If the percent recovery or the precision between the MS and MSD is outside the limits, all calculations should be checked and the data may be qualified in accordance with the applicable data validation protocol (see Section D2.1.2). Absent laboratory-specific precision and recovery criteria (generated by the laboratory as specified in SW-846 Method 8000, Section 8.0), the spiking compounds, and recovery and precision criteria specified in the current CLP statement of work (SOM01.1) in Forms 3B, 3D, and 3F will be applicable.

B5.1.4 Performance Evaluation (PE) Samples

Submission of PE samples is not currently planned, as PE samples for suitable contaminants, concentrations, and matrix were not be identified and procured. If appropriate PE sample become available and are analyzed, an addendum to the QAPP will be issued to address the analysis and evaluation PE sample results. PE samples, if utilized, may include standard reference materials (SRMs) certified by NIST. Previous data for inorganics did not suggest analytical deviations warranting the analysis of PE samples for this part of the work.

The CLP SOW for CDD/CDFs (DLM02.0) requires analysis of a LCS at a frequency of one per 20 field samples.

B5.2 Laboratory QA/QC Procedures

B5.2.1 Method Blanks

The purpose of analyzing method blanks is to demonstrate that the analytical procedures do not result in sample contamination from the laboratory solvents, reagents, or glassware used in processing the samples. Method blanks will be prepared and analyzed by the contract laboratory at a rate of at least one per analytical batch. Method blanks will consist of laboratory-prepared blank water processed along with the batch of environmental samples including all manipulations performed on actual samples. The method blank should be prepared and analyzed before analysis of the associated environmental samples. Method blank concentrations should be below the reporting limit for each target analyte. (There are exceptions for a few volatile and semivolatile organics, which may be tolerated at concentrations up to five times the reporting limit. However, none of these exceptions are target analytes for the Passaic River Pilot Dredging program. CLP SOW DLM02.0 allows up to three times the CRQL for octachlorinated dioxins and furans [OCDD and OCDF].) If the analyte concentrations in a method blank exceed the criteria, the source of contamination should be corrected, and the associated samples should be reanalyzed. If reanalysis is not possible, the laboratory will flag the associated data (B qualifier) and note the deviation in the case narrative.

Sample data will not be corrected or adjusted for blank contamination. Blank contamination will be addressed during data validation; and the data validator will determine whether the data should be accepted (typically in cases where the sample concentration is at least five times higher than the blank concentration; the B-flag is then removed by the validator) or negated (the reported concentration is flagged ‘U’ and the B-flag is removed; typically where the sample concentration is less than five times the blank concentration).

B5.2.2 Laboratory Control Samples

The purpose of analyzing laboratory control samples is to demonstrate the accuracy of the analytical method. Laboratory control samples (LCSs) will be analyzed at the rate of one per sample batch (up to 20 samples). Laboratory control spikes consist of laboratory-fortified (spiked) method blanks. The LCS is spiked with the same analytes at the same concentrations as the MS (for methods for which MS analysis is required). When the MS results indicate a potential sample matrix-related problem, the LCS results are used to confirm that the laboratory can perform the analysis in a clean matrix. In general, criteria for LCS analyses will be equal to or more stringent than those established for the corresponding MS. Each laboratory should develop its own recovery criteria; absent laboratory-generated criteria, an initial default recovery goal of 70 to 130 percent may be utilized.

For PCB congeners, a ‘native toxics/LOC standard spiking solution’ spiked into reagent water is used (for ongoing precision and recovery), as well as a QC Check Sample. The default OPR criteria are 50 to 150 percent for all 27 congeners in the spiking solution. For CDDs/CDFs, all 17 target isomers are present in the LCS and the (analyte-specific) recovery criteria range from 63 to 170 percent for the CLP method (see DLM02.0 Exhibit D, Table 6 for analyte-specific limits). If the LCS recovery not within method-specified criteria, the analytical process is not being performed adequately, and the appropriate corrective action must be undertaken (e.g., re-extraction and reanalysis of associated samples). For PCDD/PCDF analysis by Method 1613B, there is no direct analog to the LCS but its function is achieved through the analysis of the QCS and a Precision and

Accuracy (PAR) standard. If reanalysis is not possible, the associated sample results should be qualified as low or high biased. The percent recovery of the LCS is calculated by the following equation:

$$\%REC = (A)/T \times 100\%$$

Where: A = concentration of analyte in the spike sample aliquot
T = known true value of the spike concentration

B5.2.3 Temperature Blanks

The purpose of preparing temperature blanks and sending the temperature blanks in the sample coolers on location is to enable the laboratory to monitor the temperature of the coolers (and samples) upon receipt at the laboratory. A temperature blank will be provided in each cooler sent from the laboratory to the field.

If the temperature of the cooler upon receipt exceeds 6° C (10º C for CLP laboratories), the laboratory will immediately contact the client (SMO for CLP) for instructions as to how to proceed (e.g., discard the sample; or analyze as planned and note deviation in the case narrative).

B6 Equipment/Instrumentation Testing, Inspection, and Maintenance

B6.1 Field Equipment

Equipment failure will be minimized by inspecting field equipment to ascertain that it is operational and by performing appropriate preventive maintenance activities. Field sampling equipment and associated support equipment will be inspected prior to collecting each sample and any necessary repairs will be made prior to decontaminating and reusing the equipment. Routine daily maintenance procedures of field equipment to be conducted in the field will include:

- Removal of surface dirt and debris from exposed surfaces of the sampling equipment and measurement systems,
- Storage of equipment away from the elements,
- Daily inspections of sampling equipment and measurement systems for possible problems (e.g., damage or weak batteries),
- Check instrument calibrations as described in Section B7 of the QAPP, and
- Charging battery packs for equipment when not in use.

Field equipment maintenance will be documented in the applicable field logs. Specific equipment that will be inspected/tested includes:

- The Global Positioning System on the sampling vessel and the geophysical testing equipment will be maintained in accordance with the manufacturer's recommendations.

- Instrumentation used to water quality data will be maintained in accordance with the manufacturer's recommendations.
- Totalizing flow meters associate with the TOPS apparatus will be inspected daily to verify that it is in proper working condition and maintained in accordance with manufacturer's recommendations.
- Sampling vessels will be inspected daily and maintained in good working order.

Spare parts and supplies will be stored in the field to minimize downtime. These items include, but are not limited to, the following:

- Appropriately-sized batteries,
- Extra sample containers and preservatives,
- Extra sample coolers, packing material, and ice,
- Sufficient supply of decontamination materials (Alconox, distilled/deionized water)
- Distilled (analyte-free) water
- Backup ISCO samplers will be available from USGS (on site, or in Trenton),
- Additional supply of health and safety equipment (e.g., respirator cartridges, boots, gloves, Tyvek, etc.), and
- Additional equipment, as necessary, for the field tasks.

B6.2 Laboratory Instrumentation

The primary goals of the project laboratory's preventive maintenance programs will be to prevent instrument and equipment failure as much as possible and to minimize instrument down time when failure occurs. The laboratory(ies) will maintain an inventory of replacement parts needed for preventive maintenance and spare parts that routinely need replacement (e.g., septa, gauges, sources, detectors, etc.). Implementation and documentation of the preventative maintenance program will be primarily the responsibility of the technical group using the instrumentation according to the individual laboratory preventative maintenance policies in its Laboratory Quality Manual. If an instrument fails, the problem will be diagnosed as quickly as possible, and either replacement parts will be ordered or a service call will be placed to the manufacturer. If instrument failure impedes sample analysis, the QA Program Manger will be notified promptly so that appropriate corrective action and sample capacity management can occur. Preventive maintenance and maintenance performed as corrective action will be documented by the group leader, analyst, or contracted service representative who performed the procedure and the documentation will be maintained at the individual laboratory.

B7 Calibration Procedures and Frequency

B7.1 Field Instruments and Calibration

It is expected that field measurements and equipment will include, but may not be limited to, GPS readings and water quality data (CDT, LISST, and ADCP). The GPS on the sampling vessel will have a daily check on a point with known coordinates. Geophysical testing equipment will be calibrated and maintained in accordance with the manufacturer's recommendations. If these criteria are not met, appropriate adjustments/modifications will be made.

Field measurements obtained during field data collection will be collected with properly calibrated instruments. Field personnel will follow the procedures described by the manufacturer's recommendation and as described below.

In general, field instruments will be calibrated prior to use and the instrument calibration checked after the final use on each day. Personnel performing instrument calibrations will be trained in its proper operation and calibration. Equipment will be maintained and repaired in accordance with manufacturer's specifications (Section B6). In addition, prior to use, each major piece of equipment will be cleaned, decontaminated, checked for damage, and repaired, if needed. Field calibration activities will be noted in a field log notebook that will include, at a minimum, the following:

- Entries to the instrument logbooks will be made at least once daily whenever the instrument is in use.
- Calibration records will include:
  - Calibrator's name,
  - Instrument name/model,
  - Date/time of calibration,
  - calibration standard(s) used and source,
  - Temperature (if it influences the measurement),
  - Results of calibration (raw data and summary), and
  - Corrective actions taken.

The totalizing flow meters used for recording the flow through the GFF and XAD resin cartridges (and the separate flow meter for the membrane filter used to acquire the particulate sample for metals analysis, if used) in the TOPS will be assessed by comparing volumes measured with flow totalizers against masses of water measured on calibrated scales. A pair of ISCO samplers will be utilized on each of the two TOPS boats to collect discrete samples for TSS and organic carbon analysis for each transect during each three-hour sampling event (corresponding with the time that water is passing through the GFF and XAD resin for TSS analysis). Maintenance and calibration of the ISCO samplers is described in Appendix C (USGS SOP #4).

B7.2 Laboratory Analytical Instrumentation and Calibrations

Calibration of laboratory analytical instrumentation is required for the generation of appropriate data to meet project data quality objectives. Detailed calibration procedures, calibration frequency and acceptance criteria are specified in the analytical method SOPs. Each laboratory contracted for this project will be responsible for the proper calibration and maintenance of laboratory analytical equipment. Calibration activities performed will be documented in the analytical data package (see Section A9) and will be available for review during external laboratory audits.

In general, reference standards used will bracket the expected concentration of the samples. At a minimum, this generally will require the use of three to five different standard concentration levels that are used to demonstrate the instrument's linear range in quantitation. Calibration of an instrument must be performed prior to the analysis of any sample and then at periodic intervals (continuing calibration) during the sample analyses to verify that the instrument is still calibrated. Sample concentrations are often outside the instruments linear range and, therefore, need to be diluted and reanalyzed. The specific analytical methods also provide the calibration acceptance criteria and corrective actions to be employed if the acceptance criteria are not met (e.g., recalibration).

B7.3 Standard and Standards Records

Standards used by laboratories are described in the laboratory analytical methods. Laboratory standards will not be used if there are indications of physical deterioration (such as discoloration), or if the shelf life of the standard (as established by the manufacturer) is exceeded. Appropriate records of laboratory standards will be maintained in the laboratory, including the following:

- name and source,
- date received,
- lot number or manufacturer's tracking number,
- stock and initial concentration calculations, and
- storage requirements and storage location.

B8 Inspection/Acceptance Requirements of Supplies and Consumables

Only supplies and consumables that are of adequate quality to sustain confidence in the sample collection, processing, and laboratory analyses will be used for this project. Where no independent assurance of the quality of outside supplies is available (including materials provided by third parties, procedures to verify that the quality of the purchased materials are consistent with the overall project technical and quality criteria will be established. It is especially important that sample media (glass fiber filters, XAD resin cartridges; membrane filters; ultra-clean bottles) be properly prepared; and documentation verifying that the media are sufficiently contaminant-free for project purposes must be reviewed before acceptance and use of these materials. (See Appendix B for the SOP addressing decontamination and custody of specially-cleaned sample media including GFF and XAD.) Purchased supplies and consumables will not be used until they have been inspected, calibrated or otherwise verified to verify compliance with any specifications relevant to the calibrations or tests being performed for the project. Records of supplies and consumables used for the project will be maintained, including batch/lot numbers (as appropriate) for sample containers and media.

B8.1 Inspection and Acceptance Testing of Supplies and Consumables

Inspections or acceptance testing will be documented, including the procedures to be followed (including acceptance criteria and testing method), the individuals responsible, frequency of evaluation, and handling and storage conditions. The established procedures must enable project personnel to verify, prior to use, that critical supplies and consumables meet relevant project quality objectives. Supplies and consumables used during sample collection and processing will be inspected on an as-received basis. In the analytical laboratories, each analyst verifying the quality of reagents/standards must be qualified to perform the associated instrumental analysis so that they can calibrate the instrument, use the data system to establish sequences, perform calculations, and interpret the data.

B8.2 Documentation and Tracking of Supplies and Consumables

Records for purchases and receipt of supplies and consumables for sample collection and processing activities will be maintained in the field processing facility. Return of damaged or inappropriate materials to the suppliers will also be documented.

Documented procedures shall exist at each project laboratory for the purchase, receipt, handling/storage, and tracking of supplies and consumables used for the technical operations. The established procedures must enable project personnel to confirm that supplies and consumables that have not been tested, have expired, or do not meet acceptance criteria are not used for the project. Laboratory personnel performing reagent/standard preparation must have demonstrated proficiency that they understand the dilutions, preparations, and documentation required after training with a qualified person.

Each project laboratory will retain records for standards, reagents and media including the manufacturer/vendor, the manufacturer’s Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material may not be used unless its reliability is verified by the laboratory. The original containers (such as provided by the manufacturer or vendor) will be labeled with a unique identifier that links the containers to the aforementioned records, the date opened, and the expiration/reevaluation date.

Records of reagent and standard preparation will be maintained by the laboratory. These records indicate traceability to the purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date, and preparer’s initials. Containers of prepared reagents and standards must bear a unique identifier and expiration/ reevaluation date and be linked to the aforementioned records. Labels that indicate the following information are to be used for reagents and standards:

- Unique Identifier (notebook reference indicating where the reagent preparation is documented),
- Name of the material,

- Concentration,
- Date prepared,
- Storage Conditions, and
- Expiration/reevaluation date.

B9 Data Acquisition Requirements (Non-Direct Measurements)

Data such as river flow rate may be provided by others (e.g., USGS). Verifying the quality of data from outside sources is the responsibility of the data user.

Historical data were utilized as part of the criteria for the selection of the study area for this project. Preliminary unpublished data from recent investigations in the Harrison Reach of the Lower Passaic River were also reviewed qualitatively as an aid to identifying approximate analytical sensitivity needed for this project. As the historical and unpublished data were used only in a general manner (identifying approximate levels of contamination), no detailed data quality review was necessary or appropriate.

B10 Data Management

The following subsections present an overview of the project information management system. This includes the field sample data collection process, the required specifications of the data deliverables, and the storage, review, and retrieval of analytical data.

To support the storage, evaluation, and retrieval of analytical results, commercially available software such as Fox Pro, a relational database management system, will be used to organize the data. Ultimately, the data will be uploaded into the project web site (PREMis) and upon full acceptance of the data to the public web site (ourpassaic.org).

B10.1 Purpose/Background

Analytical laboratories will provide preliminary electronic data files in Excel-compatible spreadsheet form and final data also as a tab-delimited file.

The analytical laboratories will report the data based on the samples that are analyzed; the laboratory will not manipulate the data or convert it to other units. For example, the laboratory will report organics data from the GFF media as “ng/sample”. It is the responsibility of the project team to acquire the additional data and make the necessary calculations for reporting in other units (e.g., “ng/kg suspended matter”).

B10.2 Data Recording

As discussed above, the field sample data collection will be done using field books and sample logs. Forms specific to USGS operations are provided in Appendix C (USGS SOP #2).

B10.3 Data Validation

The data validation process is described in Section D2.1 of this QAPP.

The criteria for data verification are as described in the data validation SOPs in this QAPP and the evaluation process described in Section D2.1; specific procedures for validation (i.e., validation SOPs) are listed in Section D2.1.2.

In general, the actions identified below will be taken when data quality indicators do not meet method or project criteria. However, the list below is general; the specific data validation documents cited in Section D of this QAPP take precedence over the summary below.

- Data generated from samples analyzed outside of holding time criteria will be qualified as estimated (for minor exceedances) or rejected (in the event of gross exceedance).
- Organic sample data associated with surrogate recoveries greater than or less than the control limits will have values greater than the sample reporting limit qualified as estimated.
- Organic sample data associated with surrogate recoveries below the control limits but greater than or equal to 10 percent will have non-detected values qualified as estimated.
- Organic sample data with surrogate recoveries less than 10 percent will have non-detected results rejected.
- Organic sample analysis data with MS or MSD recoveries or relative percent differences (RPDs) outside of control limits will have the specific out-of-criteria compound result(s) in the associated unspiked sample qualified. Qualification for matrix spike analyses follows the QC rules used for surrogates using the associated QC Limit.
- Samples associated with (i.e., samples in the same preparation batch) LCS sample recoveries outside of criteria will be qualified following the same rules as for surrogates using the associated QC Limit.
- Inorganic spike sample analyses with recoveries outside of criteria will have all samples of the same matrix on the chain-of-custody qualified following the same rules as for surrogates using the associated QC Limit.
- Inorganic laboratory duplicate analyses not meeting the method-specified RPD criteria will have the analyte values greater than the sample reporting limit qualified as estimated in similar matrix samples in the same lab preparation batch.
- Field duplicate analytes with out-of-criteria RPDs will have the analyte values greater than the sample reporting limit estimated in only the field duplicate and its associated sample. The precision goal is RPD less than or equal to 50 percent for organic and inorganic analyses of water samples for detected values at least five times greater than the sample-specific reporting limit.

C. ASSESSMENT AND OVERSIGHT

C1 Assessments and Response Actions

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits.

C1.1 Field System Audits

C1.1.1 Internal Field System Audits Responsibilities and Frequency

Internal field system audits are those conducted by the organization conducting the work (i.e., TAMS/ET and MPI). Due to the limited scope and short duration (about five days) of the pilot dredging project, no formal internal audits of field activities including sampling and field measurements will be conducted. However, it is the responsibility of the field team leader (or designee), and ultimately the responsibility of the project manager, to perform the work in accordance with the requirements of the project; as such, the monitoring of the following items will be conducted during the execution of the pilot dredging program:

- Completeness and accuracy of sample chain-of-custody forms including documentation of times, dates, transaction descriptions, and signatures,
- Completeness and accuracy of sample identification labels including notation of time, date, location, type of sample, person collecting sample, preservation method used, and type of testing required,
- Completeness and accuracy of field notebooks or records including documentation of times, dates, subcontractor names, sampling method used, sampling locations, number of samples taken, name of person collecting samples, types of samples, results of field measurements, sample logs including identity of duplicate or co-located samples, and any problems encountered during sampling,
- Adherence to health and safety guidelines outlined in the Site Safe Work Plan (SWP), including wearing of proper personal protective equipment (PPE) and adherence with established safety protocols for working on boats and near water,
- Adherence to decontamination procedures outlined in Section B2.7 of this QAPP including proper decontamination of sampling and processing equipment, including proper storage and handling of equipment decontaminated off-site (e.g., GFF filters and XAD resin cartridges; and ultra-clean sample containers for trace mercury), and
- Adherence to sample collection (including ‘clean hands’ sampling), preparation, preservation, and storage procedures.

C1.1.2 External On-Site Field System Audits

External field audits are those conducted by organizations not affiliated with those performing the work. Such audits will be conducted at the discretion of the client and the frequency and procedures for external audits are identified by the client and are not established in this QAPP.

C1.2 Laboratory System Audits

As with field operations, laboratory systems audits may be internal or external.

C1.2.1 Internal Laboratory System Audits

Due to the limited scope and duration of the pilot dredging program, TAMS/ET and MPI will not perform project-specific laboratory systems audits. In addition, administrative issues preclude TAMS/ET and MPI personnel from auditing government-acquired laboratory services (DESA and CLP). However, each individual laboratory QA Manager performing analytical testing services for this project will perform periodic internal systems audits to evaluate laboratory operations and quality control procedures in accordance with its individual Laboratory Quality Manual. These audits are intended to serve two purposes: to verify that the laboratories are complying with the procedures defined in laboratory manuals and contracts; and to determine any sample flow or analytical problems. Internal audits performed by the participating laboratories prior to or during the time the Passaic River sample analyses are being performed may be requested by QA Program Manager as necessary to review associated QA/QC issues relevant to this project.

Laboratory services acquired through the USEPA Contract Laboratory Program (including non-RAS analyses) will be audited in accordance with the requirements of the applicable CLP statement of work and contract requirements.

C1.2.2 External Laboratory System Audits – Responsibilities and Frequency

Independent assessment of the laboratory operations will be through the audits conducted by accrediting agencies. As such, it is important that laboratories selected for this project by accredited by recognized agencies or organizations; and where possible, be certified or validated for the specific parameters being analyzed. Principle accrediting organizations for this project include USACE, NJDEP, NYSDEC/NYSDOH, and NELAC.

Laboratories in the USEPA CLP are subject to audit by USEPA as part of their participation in the CLP. (The CLP, however, is a contracting, not an accrediting, program.) The USEPA Region 2 laboratory (Edison, NJ) participates in NELAC and is subject to the audit provisions of that program.

Some of the analyses being performed for this project are not sufficiently used to have method-specific certification available (e.g., USEPA CBC01.0 for PCB congeners).

Laboratories selected for these methods will have undergone reviews of their laboratory operations in general by USEPA to confirm the competence of the laboratory for the specific method.

C1.3 Corrective Action

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or poor QC performance that can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, and data assessment. Corrective actions proposed and implemented will be documented in the regular quality assurance reports to management (Section C2). Corrective action will only be implemented after approval by the Project Manager or his designee. If immediate corrective action is required, approvals secured by telephone from the Project Manager will be documented (e.g., in a memorandum).

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Project Manager, who in turn will assess whether the non-conformance is of sufficient significance to warrant immediate notification of the client (NJDOT-OMR).

Any nonconformance with the established QC procedures in the QAPP will be identified and corrected in accordance with the QAPP. The Project Manager, or his designee, will issue a nonconformance report for each nonconformance condition.

C1.3.1 Field Corrective Action – Water Sampling Program

Corrective action in the field may be initiated when the sample network or rationale is changed (e.g., changes to sample quantities or sampling locations from those specified in the QAPP), or when sampling or field analytical procedures require modification due to unexpected conditions. In general, the field team may identify the need for corrective action. The field staff, in consultation and concurrence with the Project Manager and Agency PM (if practical), will recommend a corrective action. The Project Manager will approve the corrective measure that will be implemented by the field team. It will be the responsibility of the Field Team Leader to verify that the corrective action has been implemented.

If the corrective action will supplement the existing sampling plan (e.g., additional samples) using existing procedures as specified in this QAPP, corrective action approved by the Field Team Leader will be documented. If corrective actions resulting in fewer samples (or analytical fractions), alternate locations, etc., compromise the achievement of quality assurance objectives, it will be necessary that all levels of project management, including Project Manager and the NJDOT-OMR, concur with the proposed action.

C1.3.2 Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during, and after initial analyses. The published methods and/or laboratory SOPs specify the majority of the conditions during or after analysis that automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, or automatic reinjection/reanalysis when certain QC criteria are not met. Furthermore, a number of conditions, such as broken or leaking sample containers, insufficient sample volume, multiple phases, low/high pH readings, and potentially high concentration samples, may be identified during sample log-in or just prior to analysis. Following consultation with laboratory analysts, it may be necessary for the laboratory QA Officer to approve the implementation of corrective action.

A member of the laboratory technical staff will identify the need for corrective action. The laboratory QA Officer, in consultation with members of the technical staff, will approve the required corrective action to be implemented by designated members of the laboratory technical staff. The laboratory QA Officer is responsible for the implementation and documentation of the corrective action. If the nonconformance causes project objectives not to be achieved, it will be necessary to inform the QA Program Manager who must concur with the corrective action.

Corrective actions that are performed prior to release of the data from the laboratory will be documented in a laboratory corrective action log and in the narrative data report that will be included in the laboratory data deliverable package. If corrective action does not rectify the situation, the laboratory will contact the QA Program Manager prior to release of the data.

C1.3.3 Corrective Action During Data Validation and Data Assessment

The need for corrective action may be identified during the data verification, data validation or data assessment process. Potential types of corrective action may include resampling by the field team (although this is unlikely to be feasible due to the limited scope and duration of this project) or reinjection/reanalysis of samples by the laboratory.

As previously stated (Section A7.3.5), the percent completeness will be used to determine whether the data quality meets the objectives for the project. If the completeness objectives are not met for individual parameters, the QA Program Manager will review the reasons for the incomplete data with the Project Manager. Depending on the reasons for the incomplete data (e.g., holding time exceeded), and the effect of the incomplete data on the accomplishment of the project objectives, additional samples may be collected and analyzed. An evaluation will also be conducted if a sample does not generate data for a parameter category (e.g., PCB congeners, metals). Such a data gap could result from sample container breakage or sample loss during analysis. If it is determined that the missing results are critical to accomplishing the work plan objectives, additional sampling may be conducted to obtain the missing data (although this is unlikely to be a viable option for this project). The Project Manager and NJDOT-OMR will be responsible for approving the implementation of corrective action, including resampling, during data assessment. The QA Program Manager will document corrective actions of this type.

C2 Reports to Management

Periodic progress reports will be submitted to the client, and will include a summary of quality assurance and quality control activities that occurred during the reporting period. The QA portion of the monthly progress reports will be the responsibility of the QA Program Manager and will provide a status report on the accuracy, precision, and completeness of the data as well as the results of the performance and system audits, and corrective action needed or taken during the project.

C2.1 Contents of the QA Section of the Monthly Reports

The QA section of the monthly progress reports will contain results of field and laboratory audits performed during the reporting period, information generated during the reporting period reflecting on the achievement of specific DQOs (including data validation and assessment results), and a summary of corrective action that was implemented and the corrective action's immediate results on the project. The status of analytical, data verification and data validation tasks will be summarized for the project with respect to the project schedule. In addition, whenever necessary, updates on training provided, changes in key personnel, and anticipated problems in the field or laboratory for the coming reporting period that could bear on data quality along with proposed solutions will be reported. Progress reports will be prepared in written, final form by the Project Manager or his designee. To the extent possible, the monthly reports will include an assessment of the project will also be performed on the basis of available QC data and overall results in relation to the planned objectives.

C2.2 Frequency of QA Reports

The QA summary will be prepared as part of the required monthly progress report. The reports will continue until the project has been completed, unless an alternate reporting scheme is agreed to by the client (NJDOT-OMR).

In the event of an emergency, or in case it is essential to implement corrective action immediately, QA reports can be made by telephone to the appropriate individuals, as identified in the Corrective Action sections of this QAPP (C1.3). These events and their resolution will be addressed thoroughly in the next issue of the monthly progress report.

C2.3 Individual Receiving/Reviewing QA Reports

Copies of the monthly report containing the summary of QA activities will be submitted by TAMS/ET to NJDOT-OMR. NJDOT-OMR will be responsible for distribution of copies to the appropriate organizations and individuals. Additional project team members will receive the monthly progress report on a 'need-to-know' basis.

D. DATA VALIDATION AND USABILITY

The QA procedures that will occur after data collection are described in this section.

D1 Data Review, Assessment, and Validation

The field, laboratory, and data management activities described in this QAPP will be reviewed to assess whether these activities were performed in a manner that is appropriate for accomplishing the project objectives. This assessment will include electronic data assessment, followed by data validation. Assessment of the data is performed to determine whether the data have been generated in accordance with the specified procedures as established in analytical SOPs and the QAPP. Data validation involves identifying the technical usability of the data for making decisions pertaining to satisfying the project objectives identified in Section A7.

D1.1 Review of Sampling Design

The project manager or designee will review the collected samples to verify conformance with to the sampling design specifications in Section B1 of the QAPP. Samples that deviate from the sampling design and the impact to project objectives, if any, will be discussed in the final report prepared at the end of this pilot dredging program.

D1.2 Review of Sample Collection Procedures

The sample collection procedures employed by the sampling personnel be reviewed to confirm that the samples are collected in accordance with the procedures identified in Section B2 of this QAPP. This review will note unacceptable departures, if any, from sample collection procedures in the QAPP and identify sample data (analytical or field) that should be excluded from incorporation into the project database or data evaluation process. In addition, the Field Team Leader or his designee will review project logbooks or records at the conclusion of sampling activities.

As this project is of short duration (five days), no formal field audit will be performed during sample collection.

The evaluation (data review) of equipment blanks and other field QC samples will provide definitive indications of the data quality. If a problem arises, it should be able to be isolated via the complete sample tracking and documentation procedures that will be performed. If such a problem does arise, corrective action can be instituted and documented. If data are compromised due to a problem, appropriate data qualifications will be used to identify the data. The labeling and identification of samples will also be reviewed to confirm that samples properly represent the location they were intended to represent.

D1.3 Review of Sample Handling

The handling, preservation and storage of samples collected during the sampling program will be monitored on an on-going basis. Additionally, the project laboratories will document sample receipt including proper containers and preservation at the time samples are logged into their individual laboratory. The sample receipt records (a required data package deliverable) as well as traffic report/chain-of-custody documentation will be routinely assessed by the data validators during data validation. Sample handling, storage, or preservation problems identified during data validation will result in appropriate qualification of data to warn the data user to data quality deficiencies or potential mis-identification of samples.

D1.4 Review of Analytical Procedures

The use of the proper analytical procedures described in Section B4 of the QAPP will be reviewed primarily through the data verification and data validation methods discussed in Section D2 of this QAPP. Qualification of data that does not conform to criteria is also discussed in Section D2 of this QAPP. Confirmation that samples were analyzed for the proper analyses will be performed to determine if samples submitted for analysis actually had the analyses performed. If analyses that were identified to be performed were not actually performed (e.g., due to loss of sample or improper log-in at the laboratory) then a determination should have been made at the time the missing data was discovered and appropriate corrective action documented. The project manager will review the impact of incomplete analyses and identify impacts to the project objectives, if any, in the final project report.

D1.5 Review of Quality Control

The review of quality control checks described in Section B5 of the QAPP will be reviewed primarily through the data verification and data validation methods discussed in Section D2 of this QAPP. Qualification of data that does not conform to criteria is also discussed in Section D2 of this QAPP.

D1.6 Review of Calibration

The review of calibration of instruments and equipment described in Section B7 of the QAPP will be reviewed primarily through the data verification and data validation methods discussed in Section D2 of this QAPP. Qualification of data that does not conform to criteria is also discussed in Section D2 of this QAPP. The Field Team Leader will review records of field equipment calibration and identify any impacts to non-analytical data that may exist.

D1.7 Data Reduction and Processing

Data generated through field activities or by laboratory operations will be reduced and reviewed or validated prior to reporting. The field contractor or laboratory will not disseminate data until it has been subjected to these reduction and internal validation procedures that are summarized in subsections below.

D1.7.1 Data Reduction

Data reduction involves the process of generating qualitative and quantitative sample information through observations, field procedures, analytical measurements, and calculations.

Data reduction occurs with:

- The QAPP through sample locations and naming conventions;
- The field sampling process through use of field logs and field measurements;
- Communications with the laboratory in sample analysis requests;
- Field operations with collection, preservation, and chain-of-custody documentation;
- Laboratory operations with sample receipt and handling, sample-specific field data (e.g., flow data for GFF and XAD cartridge samples); sample preparation and analysis, collation of raw data, and generation of laboratory results;
- Data processing to convert ‘as-measured’ data to the final desired units (laboratory results on XAD cartridges and GFF filters, reported on a mass/sample basis, to mass per volume of water); and
- Post-laboratory operations with collation of analytical results in a format suitable for documents such as reports and maps.

Data reduction steps include field operations, laboratory operations, and report preparation operations.

Specific QC measures developed to maintain accuracy throughout the data reduction process are described throughout this QAPP.

D1.7.1.1 Field Sampling Data Reduction Procedures

Field data will be recorded manually on a field log sheet at the time of core collection. These data will include:

- Date
- Time
- Sample identification
- Horizontal coordinates (northing, easting)
- Water surface elevation
- Water depth
- Depth within water column at which sample was collected
- Flow rate/time/volume (for GFF and XAD cartridge samples)
- Field observations
- Any additional information entered into the field database.

If errors are made on the field logs, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. The field logs will be reviewed at the end of each day.

D1.7.1.2 Laboratory Data Reduction Procedures

Laboratory data reduction procedures will be followed according to the following protocol. Raw analytical data will be recorded in the individual laboratory's Laboratory Information Management System (LIMS) (or equivalent) and tabular summary tables will be generated. Other pertinent information, such as the sample identification number, the analytical method used, the name of the analyst, the date of analysis, and matrix sampled will also be recorded in LIMS. At a minimum, reagent concentrations, instrument settings, and raw data will be recorded and retained in hard copy and laboratory notebooks, which are signed and dated by the analyst. The laboratory will maintain (file) copies of instrument printouts (such as gas chromatograms). Periodic review of raw data and of the computerized records by the laboratory personnel will occur prior to final data reporting according to each laboratory's Laboratory Quality Manual.

For this project, the equations that will be employed in reducing data are presented in the laboratory SOPs. (In addition, several of these equations, expressing analytical accuracy and precision, have been presented in Sections A7.3 and B5 of this QAPP.) The laboratory technical staff will check calculations prior to the release of the data. Errors will be noted, and corrections will be made. The original notations will be crossed out legibly. Analytical results for sediment samples will be calculated and reported on a dry-weight basis.

Quality control data (e.g., laboratory duplicate results, surrogate recoveries, matrix spike recoveries, and matrix spike duplicate recoveries) will be compared to the acceptance criteria. Acceptance criteria will be either the specific numeric values in the method (e.g., CLP non-RAS SOWs); laboratory-generated performance criteria or other approved criteria in the laboratory analytical SOP; or generic default criteria for the method or parameter type (e.g., SW-846 generic recovery criteria in Method 8000).

Data considered to be acceptable will be entered into the laboratory computer system. Data summaries will be sent to the laboratory Quality Assurance Officer for review. Unacceptable data will be appropriately qualified in the project report. The laboratory will prepare case narratives that will include information concerning data that was outside acceptance limits and any other anomalous conditions encountered during sample analysis. After the laboratory Quality Assurance Officer approves these data, the data are considered ready for release to the Passaic River project team.

D1.7.2 Identification and Treatment of Outliers

Outliers are unusually large or unusually small values in a population of observations. Outliers may be the result of a variety of circumstances (field or laboratory related), including any of the following:

- Sampling artifact,
- Sample integrity problem,
- Sample identification incorrectly transcribed in the field or laboratory,
- Unique conditions,
- Faulty or defective instruments,
- Inaccurate reading of meters,
- Errors in recording of data,
- Calculation errors, or
- Analytical errors.

Procedures for the identification of outliers will be followed at the data reduction stage. Outliers in laboratory data can arise from errors in analysis or from site-specific conditions that are out of the control of the laboratory. Errors in the laboratory are most often detected in the data review and validation process. In the event that quality control processes detect a suspected outlier not identified during data verification or validation, the suspect data will be subjected to appropriate statistically outlier testing. Outliers will be reported, but may not be used for evaluation purposes.

The QA Program Manager will identify outliers at the data reduction stage. When any particular value is suspected to be an outlier, the following steps will be taken:

- Other data from the same sample will be checked to see if they are also anomalous.
- The QA Program Manager will seek input from any individuals involved in generating the anomalous value as to possible causes. This will include questioning the field crew and the analyst(s).
- Field crew - If samplers demonstrate standard competency in the sampling procedure used at the time the sample with the anomalous value was obtained, sampling errors will be dismissed as a possible cause of the outlier.
- Analyst(s) - The analyst(s) will be asked to examine his/her notes and calculations and, if possible, to rerun the sample for the specific parameter in question.

Results of any samples re-extracted or re-analyzed outside holding time will be used for comparative purposes. Rejection of any suspect data or outlier will only be done by the Project Manager in conjunction with the QA Program Manager. Data may be rejected as an unacceptable outlier if:

- A problem with equipment or an incorrect procedure used during the sampling event is identified, or
- The rerun by the analyst generates a value that significantly differs from the value being examined.

D1.7.3 Data Processing

Data will be processed from verified or validated data for use in decision-making. It is expected that summary tables, maps and charts of verified and validated data will be prepared by various project team members. Data that is processed will be checked by an individual knowledgeable about the data type being compiled who will perform a check on a reasonable subset (typically about 10 percent) of the final tabulated information to verify that transcription errors have not occurred.

Further checks of the tabulated data will occur if problems are encountered or if a systematic problem is detected in the process. Systematic problems will be identified and corrected prior to processing the data again.

D2 Data Assessment and Validation Methods

Data assessment verification and data validation are conducted after samples have been collected and laboratory analytical data has been reported. Data assessment for CLP is performed by USEPA contractor personnel (SMO). Validation provides an understanding of the data quality at the completion of the sediment coring program. If correctable data quality issues are discovered, the findings must be immediately provided so that appropriate corrective action can be taken to correct or prevent the problem from recurring. The data validation occurs after the formal laboratory reports are submitted. Any issues identified during EPA data verification or data user review of preliminary results will be communicated to the USEPA Region 2 data validation coordinator.

Analytical results from commercial laboratories will also be provided in a full data package (see QAPP Section A9) in a scanned electronic media (e.g., as a portable document file [pdf]). Data from CLP laboratories will be provided in contract-required formats. Electronic deliverables will be requested from the USEPA Region 2 DESA laboratory.

Data assessment is performed on all data generated through the USEPA CLP using EPA's Data Assessment Tool (DAT). The DAT incorporates Contract Compliance Screening (CCS), Computer Assisted Data Review and Evaluation (CADRE), and Data Assessment Rapid Transmittal (DART). It provides data in electronic formats that can be uploaded into databases and provides a complete CLP data assessment package. The DAT process is described in greater detail in Section D2.2.2, below.

This data assessment process will provide an understanding of the data quality based on those QC indicators that have the most influence on qualification of data. The DAT process will be conducted upon receipt of the data so that quality of the data can be determined soon after the laboratory reports it (within 24 to 48 hours of receipt). DAT does not address data usability, qualification based upon professional judgment, suitability of data for its intended use, or compliance with the site-specific QAPP.

Full data validation will be performed by USEPA Region 2 staff or contractors on the analytical data generated through the CLP program. Data generated by the USEPA

Region 2 DESA laboratory is reviewed internally and is considered 'EPA-validated' on submission.

D2.1 Data Validation

Data validation is the process of verifying that qualitative and quantitative information generated relative to a given sample is complete and accurate. Data validation procedures will be performed for both field and laboratory operations as described below.

D2.1.1 Procedures Used to Evaluate Field Data

Procedures to evaluate field data for this program primarily include reviewing field logbooks to check for transcription errors by the field team. These procedures are performed to verify that field measurements and various quality control analyses were properly performed and documented. The field data documented includes data generated during measurement of field parameters, observations, results of any quality control sample analyses, and field instrument calibrations. This task will be the responsibility of the Field Team Leader or designee, who will otherwise not participate in making any of the field measurements or in adding notes, data, or other information to the logbook or record form.

D2.1.2 Procedures to Validate Laboratory Data

For the laboratory chemical analytical data, validation will be performed on the analytical results in accordance with the following frequencies:

- PCDDs/PDCFs by CLP SOW DLM02.0
    - Validation of all of the data (expected to be one SDG) to provide confirmation that the analyses were performed according to the requirements in the QAPP.
- Metals (TAL – ICP-MS [iron by OES], mercury by EPA 1631E)
    - Validation of metals data to confirm that the analyses were performed in accordance to the requirements in the QAPP. All data generated through the CLP are subject to validation by USEPA Region 2 staff or contractors. Data generated by the USEAP Region 2 DESA laboratory are considered EPA-validated (documented as such on the transmittal letter from DESA) and are not subject to further review.
- TCL Pesticides by SOM01.1, Part D – Pesticides/Aroclors (or CLP OLM04.3)
    - Validation of pesticides data (aroclor analysis will not be performed) will occur in order to provide confirmation that the analyses were performed in accordance to the requirements in the QAPP.
- PCBs (congeners) by CLP SOW CBC01.0
    - Validation of all (100 percent) of the PCB congener will occur in order to provide confirmation that the analyses were performed in accordance to the requirements in the QAPP, and that congener reporting (including

identification and quantitation of coeluting congeners) is performed in accordance with the requirement in the QAPP.

- TOC by USEPA Region 2 (DESA SOPs C-83 and C-88)
  - If performed by USEPA Region 2 DESA laboratory, as is currently planned, the data are validated internally by the USEPA DESA laboratory, and considered EPA-validated as submitted. No further EPA or third-party validation will be performed.
  - If performed by a laboratory other than DESA (e.g., USGS), a limited data review will be conducted, focusing on the laboratory-QC information; field duplicate precision; and reasonableness. A memorandum will be prepared documenting the scope and conclusions of the review.
- Bromide/chloride (Br/Cl) by USEPA Method 300.0.
  - It is currently planned that these analyses will be conducted by the USEPA DESA laboratory. Data generated by the USEAP Region 2 DESA laboratory are validated internally; these data are considered EPA-validated (documented as such on the transmittal letter from DESA) and are not subject to further review.
- Total Suspended Solids by DESA SOP C-33 (based on USEPA MCAWW Method 160.2
  - Formal validation of TSS data will not be conducted. The data are validated internally by the USEPA DESA laboratory, and considered EPA-validated as submitted. No further EPA or third-party validation will be performed.

The data validation strategy is based upon the fact that 100 percent verification and validation of chemical analytical data (pesticides, CDD/CDFs, PCB congeners, and metals) will occur.

Qualification of data resulting from the verification or validation processes will be reflected by assigning the appropriate qualifier code to the sample result in the project database.

The validation of the laboratory data will be performed with guidance from the USEPA Region 2 Standard Operating Procedures for the Validation of Organic and Inorganic Data (various SOPs and issue dates), supplemented as necessary by the USEPA National Functional Guidelines. These documents provide the criteria by which data are accepted, accepted with qualification, or rejected are listed below. Specific documents applicable to this project include:

- Organic data generated through the CLP program (pesticides) will be validated using SOP HW-6, Revision 11 (June 1996).
- Validation of metals data generated through the CLP program or similar methods will be by SOP HW-2, Revision 11, January 1992: Evaluation of Metals Data for the CLP Program. The USEPA National Functional Guidelines for Inorganic Data Review (October, 2004) will be the primary guidance for data generated using ICP-MS methods. Note that the metals data generated by DESA are validated

internally and are considered EPA validated and fully usable upon receipt; these data are not subject to further review.

- Validation of PCDD/PCDF data generated by DLM02.0 or by EPA method 1613B will utilize USEPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Review (August 2002). This may be supplemented as necessary with guidance from HW-25, Revision 2, September 1999: Validating Tetra - through Octa - chlorinated Dioxins and Furans by Isotope Dilution (HRGC/HRMS) by Method 1613, Revision A.
- Validation of PCB Congener Data Generated by CLP SOW CBC01.0. There is no current USEPA Region 2 SOP for validation of this method. The USEPA Region 1 SOP (Standard Operating Procedure for Toxic PCB Congeners and Total Homologue Data Validation, Revision 2, August 2001) will be used as the starting point for validation of these data. In addition, the validation process will utilize applicable portions of criteria from methods with some similarity (for guidance on how to qualify the data) in conjunction with the published method and the laboratory's analytical SOP (for guidance on the method-specific criteria which should be achieved).
- Validation of TOC data. If these data are generated by the Region 2 EPA DESA laboratory, the data are considered 'EPA-validated' when submitted by the laboratory.

The validation SOPs and guideline above will be supplemented, as appropriate, by the analytical method documents, laboratory SOPs, and the following guidance:

- USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (October 1999), and
- USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (October 2004).

These data validation SOPs provide the specific criteria used to validate the data for each analytical parameter for the project. Full validation will include an evaluation of documented QA/QC measures through a review of tabulated QC summary forms and raw instrument data.

Except as noted above, USEPA Region 2 staff or contractors will be responsible for data validation. All data analyzed through the CLP contracting system will be subject to full validation by USEPA.

D2.1.3 Data Qualifiers

Based upon the quality assurance review of the analytical data, specific codes (data qualifiers or 'flags') will be placed next to results in the database to provide and indication of the quantitative and qualitative reliability of the results. These defined qualifier codes will serve as an indication of qualitative and quantitative reliability. The data qualifier codes and definitions will be as follows:

U - The compound/analyte was analyzed for, but was not detected above the reported sample quantitation/detection limit. This applies to both samples in which the sample was reported as not detected by the laboratory, as well as compound/analytes which are considered “not detected” since it was detected in a blank at a similar level, as determined during the data quality review/data validation process.

J - Quantitation is approximate (estimated) due to limitations identified during the quality assurance review (data validation). This qualifier is applied to all data which were reported as detected at a concentration outside the limits of the calibrated range of the analysis, as well as for other reasons (minor deviations from QA/QC criteria) as determined during the data quality review/data validation process). The current version of the National Functional Guidelines for Inorganic Data Review (USEPA, 2004) also include qualifiers “J+”, estimated value with possible high bias, and “J-”, estimated value with possible low bias.

N - The analysis indicates that there is presumptive evidence to make a “tentative identification” of this compound/analyte. This flag is applicable only to organic analyses and is applied by the laboratory when an analyte does not meet all of the specified criteria for confident identification of the analyte, but is believed to be present based on the analyst’s judgment.

R - Unusable (rejected) result – compound/analyte may or may not be present in this sample.

UJ - This compound/analyte was not detected, but the quantitation/detection limit is approximate and may be inaccurate or imprecise due to QA/QC issues identified during the quality assurance review.

EMPC – (dioxin analyses only). Estimated Maximum Possible Concentration; chromatographic peaks are present in the expected retention time window, but, the peaks do not meet all of the conditions required for a positive identification. The reported result represents the estimated maximum possible concentration if the PCDD or PCDF was present.

Additional qualifiers may be present on data generated by the USEPA Region 2 DESA laboratory; such qualifiers will be defined in the DESA laboratory deliverable.

D2.1.4 Data Quality Review of Alternate or Contingency Analytical Methods

As noted previously, the analytical methods specified in this QAPP are tentative; there may be alternate methods or laboratories utilized based on technical considerations (e.g., ability of methods or laboratories to meet analytical sensitivity requirements) or resource allocation considerations (including administrative, funding and capacity issues). Use of methods not specifically addressed in this QAPP will be documented in addenda to this

QAPP, which will include both the method itself (including necessary sample preparation steps) for any method that is not a readily available USEPA method, as well as the criteria and procedures for assessing the quality of data generated by that method.

Potential alternate or contingency methods or facilities include (but are not limited to):

- Utilization of USGS analytical facilities for organic carbon and/or TSS analyses. If USGS laboratories are utilized, the analytical SOPs for the labs will be reviewed to confirm that the methods are equivalent to those proposed in this QAPP. Review of TSS data generated by USGS will be the same as described above in Section D2.1.2. If a USGS laboratory (or any non-EPA laboratory) is used to generate the organic carbon data (regardless of whether the USEPA Region 2 L Kahn method is utilized), a data quality review protocol will be established. As organic carbon analysis is largely an automated instrumental analysis, it is anticipated that the review will consist largely of verify the reported input and output values, and verifying that the analytical QA/QC specified in the laboratory's SOP and manufacturer's guidance have been adhered to.
- Utilization of trace level (modified "ultra-clean" or "clean hands") sampling and analysis methods (modified) for dissolved mercury in water for improved analytical sensitivity (e.g., USEPA Methods 1669 for sampling; 1631E for mercury analysis). Data quality review will be based on general guidance for metals analysis (e.g., National Functional Guidelines) and method-specific requirements; special attention will be paid to blank data and the potential for blank contamination to adversely affect the usability of the data.

D2.2 Procedures for Data Assessment (CLP)

Data assessment will be performed on all laboratory chemical analytical data generated through the CLP program. The five steps of the automated data assessment process, using the DAT process, are:

1. Contracted laboratories provide analytical data electronically.

2. The electronic data is processed through CCS Initial Assessment (IA) checks to determine if the data is complete and in the proper format.

3. Laboratory electronic files that pass CCS IA are processed using the EPA mainframe to generate customized electronic spreadsheets and database files (DBFs). The spreadsheet information is based on the laboratory-qualified data. The results are forwarded to Regions via the DART system, an Internet-based email system.

4. Files that pass CCS IA are loaded and processed through the CLP's data review and evaluation system.

5. The assessment results are processed using the EPA mainframe to generate customized electronic files and spreadsheets. Results from the assessment process are generated in soft copy. All files, spreadsheets/DBFs, and reports are electronically transmitted to clients through the CLP DART.

D2.2.1 CADRE

The Computer-Aided Data Review and Evaluation (CADRE) system examines Quality Control (QC) data for all analytical results and evaluates them against data review criteria that are appropriate for the corresponding analytical method/procedure and the intended use of the results. The CADRE system has predefined CLP method information and data review/QC criteria. However, it can be customized to support variations on the analytical techniques employed by the CLP and to review data according to project-specific QC criteria. CADRE uses both Regional and National Functional Guidelines to review and evaluate analytical data.

D2.2.2 CCS

The Contract Compliance Screening (CCS) system is a data assessment tool used to determine whether a laboratory has complied with all CLP Statement of Work (SOW) terms and conditions included in its contract. CCS results provide statistical and detailed reports (i.e., error messages and structural defects) on the administrative and technical defects and contributes to payment determination for the analytical services provided by a laboratory.

D.2.2.3 DART

The Data Assessment Rapid Transmittal (DART) system is an automated email system designed to distribute specified reports to specific data recipients. DART is utilized to distribute CCS and CADRE reports to specified EPA Regional contacts. In addition, the DART distribution system delivers laboratory analytical data, in spreadsheet or database format.

D2.3 Data Evaluation and Summary Report

TAMS/ET and MPI will interpret and evaluate the data and prepare summary reports that document the work performed during the pilot dredging program. After the laboratory data are available, TAMS/ET and MPI will interpret, evaluate, summarize, and tabulate the data collected during the sediment core collection effort and present the major findings to NJDOT-OMR, USEPA, and USACE. TAMS/ET and MPI will revise these summary reports to incorporate comments provided by NJDOT-OMR, USACE, EPA and other government agencies. TAMS/ET and MPI will prepare these reports in a format that will allow for easy incorporation into pertinent PMP documents. It should be noted that the current scope of work does not cover the preparation of a USACE-type Quality Control Summary Report.

D3 Reconciliation with Data Quality Objectives

The QA Program Manager in conjunction with the Project Manager will determine whether field and analytical data or data sets meet the requirements necessary for decision-making. The results of measurements will be compared to the DQO requirements set forth in this QAPP. As data are evaluated, anomalies in the data or data gaps may become apparent to the data users. Data that do not meet the DQOs will be identified and appropriately noted in the project database so the data users are aware of any limitations or concerns with the usability of the data.

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Table A-1
Project Schedule for Environmental Dredging Pilot Study

Activity	Date
Submit Draft Project Plans	June 17, 2005
Receive Comments from Agencies	July - August, 2005
Submit Final Project Plans	November 22, 2005
Receive Dredging Contractor Bids	September, 2005
Award Dredging Contract	September 30, 2005
Initiate Pilot Study (field work)	November 30 – December 12, 2005
Submit Draft Study report	Spring 2006

TABLE B-1
Lower Passaic River Environmental Dredging Pilot Dredging Study Quality Assurance Project Plan
SAMPLE BOTTLE, VOLUME, PRESERVATION, AND HOLDING TIME SUMMARY

		Sample Bottles (1)				Minimum	Preservation	Holding Time (3, 4)		
MATRIX/ANALYSIS	Analytical Method	Mat'l	Size	Qty	Source	Vol Rqd	(2)	Extraction	Analysis	Comment
GFF/XAD samples from TOPS [8]										
Pesticides - GFF only	SOM01.1 or OLM04.3-Pest	GFF filters	(Note 10)	1 set (5)	USGS/STL	NA	None	7 days	40 days	Extraction must be completed within 7 days
PCBs- Congeners - GFF only	CLP CBC01.0	"	"	"	"	NA	None	1 year	30 days (4)	CBC01.0 does not provide HT from extraction to analysis.
PCDD/PCDFs - GFF only	CLP DLM02.0	"	"	"	"	NA	None	10 days	30 days	Holding times based on CLP SOW DLM02.0 for soil samples.
Pesticides - XAD only	SOM01.1 or OLM04.3-Pest	XAD cartridges	(Note 10)	1 pair (5)	USGS/STL	NA	None	7 days	40 days	Extraction must be completed within 7 days
PCBs- Congeners - XAD only	CLP CBC01.0	"	"	"	"	NA	None	1 year	40 days (4)	CBC01.0 does not provide HT from extraction to analysis.
PCDD/PCDFs - XAD only	CLP DLM02.0	"	"	"	"	NA	None	10 days	30 days (4)	Holding times based on CLP SOW DLM02.0 for soil samples.
Metals - Whole Water										
TAL (ICP-MS inc Fe and Al)[9]	ICP MS (OES for Fe)	FP	2.5 L	1	STL	250 mL	HNO_3 to pH < 2	NA	6 months	Requires lower CRQL than in SOW
Mercury	EPA 1631 E	Glass [12]	2.5 L	1	Lab	100 mL [12]	None	NA	28 days	Clean bottles to be provided by laboratory
Metals - Dissolved (field filtered)										
TAL (ICP-MS inc Al and Fe) [9]	ICP MS (OES for Fe)	FP	2.5 L	1	STL	250	HNO_3 to pH < 2	NA	6 months	Preserve after filtering. Requires lower CRQL than in SOW
Mercury (filtered at laboratory)	EPA 1631 E	Glass [12]	NA	NA	Lab	100 mL [12]	None	NA	28 days [13]	Clean bottles and filter to be provided by laboratory
Water Samples - ISCO sampler										
Particulate Organic Carbon (11)	DESA SOP C-88	GF Filter	25 mm	3	USGS	0.5 g	None	N/A	28 Days	Filtered at USGS; TOC analysis of suspended matter on filter
Dissolved Organic Carbon (11)	DESA SOP C-83	Plastic (6)	60 mL (7)	1	USGS	60 mL	Acid to pH < 2	N/A	28 Days	Acidify after filtration (H_2SO_4)
Total Suspended Solids	DESA SOP C-33	Plastic (6)	1 L	1	USGS	100 mL	None	N/A	7 Days	USGS SOP may also be used (as backup)
Chloride and Bromide	EPA 300.0 (IC)	P or G	60 mL (6)	1	USGS	60 mL	None	N/A	28 days	50 mL for Cl, 100 for Br (MCAWW); actual reqmt is less.
Water Samples - L and M Boat Grabs										
Total Suspended Solids	DESA SOP C-33	P/G	1 L	1	USGS	100 mL	None	N/A	7 Days	DESA SOP based on EPA 160.2

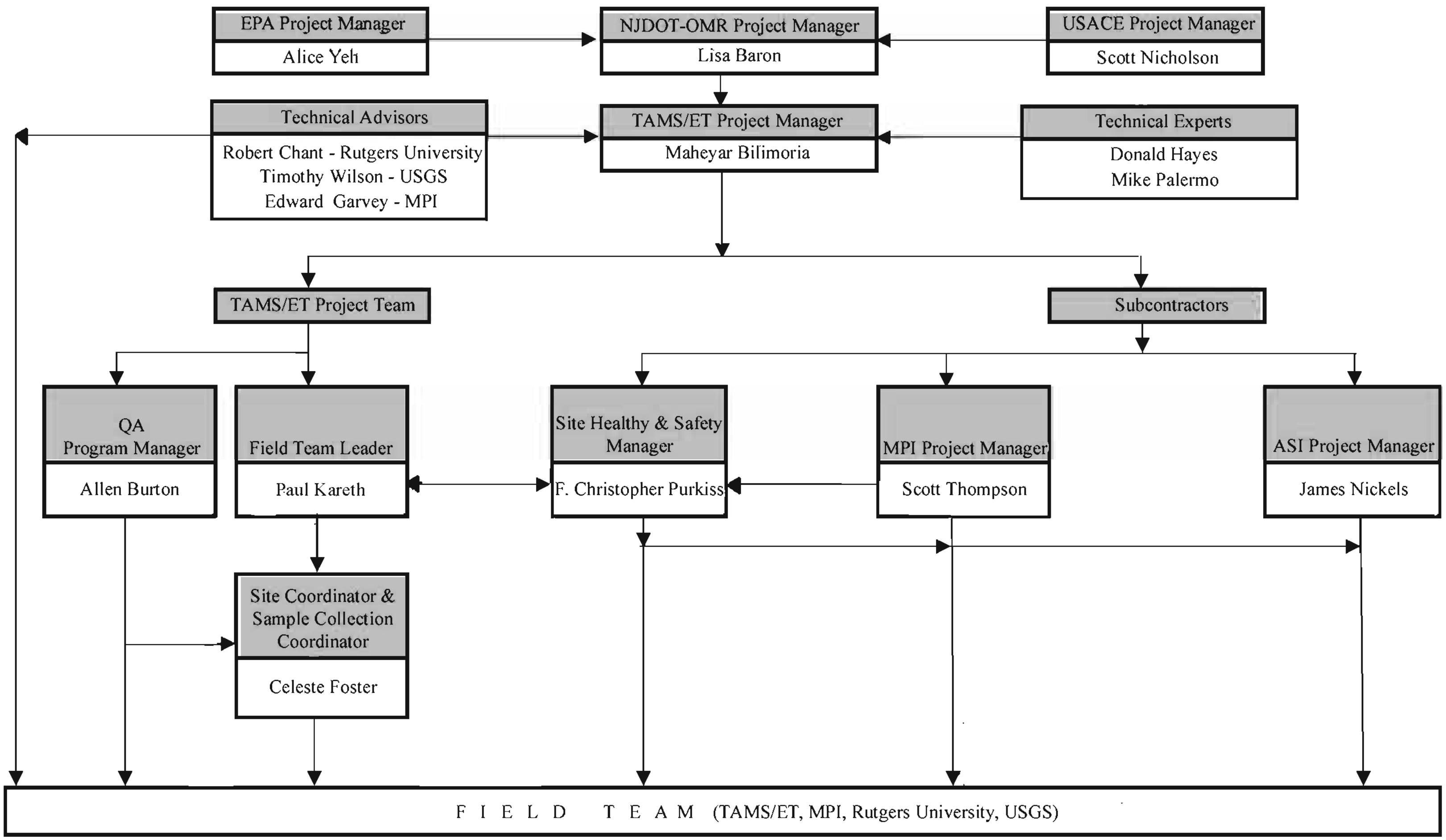
(1) Bottles shown for CLP SOW analyses as specified in USEPA, 2004. Others are typical and specific sizes and sample groupings may vary depending on laboratory-specific requirements.
(2) All samples for chemical analysis should be held at 4 degrees C in addition to any chemical preservation required.
(3) Holding time for calculated from day of collection.
(4) Samples and extracts may at less than 6 deg C for up to one year; Soil samples are stored at less than -10 deg C per CBC01.0. Holding time after extraction is default; none specified in CBC01.0..
(5) One set consists of two glass fiber filters (GFF), or two XAD resin cartridges. The GFF and XAD are extracted and analyzed separately as shown in Table 2.
(6) ISCO sample bottles are wedge-shaped plastic designed specifically to fit into the ISCO sampler. These bottles in which the sample is collected will also be used to ship the sample to the analytical or processing laboratory.
(7) TOC (if needed) may be calculated from DOC and particulate organic carbon.
(8) GFF (glass fiber filters) and XAD Resin cartridges analyzed separately but the same QC requirements apply to each. Project-specific single extraction performed for all fractions for each media (GFF and XAD).
(9) Analyte list to include all metals amenable to analysis by ICP-MS including Al and Fe. Specific QC requirements are shown for lead. Fe analysis by ICP-OES (RL = 100 ug/L). RL for Al is 60 ug/L.
(10) GFF sample consists of one flat filter and one cartridge filter. XAD sample consists of one pair of resin cartridges (each 4" by 2" diameter).
(11) POC and DOC are collected as whole water and transported to the USGS lab in Trenton where the sample is filtered and the filters and filtered water are generated for POC and DOC analysis.
(12) Tentatively assigned laboratory (STL, North Canton) will utilize one 2.5-L single-use bottle for low-level Hg analysis; aliquot of sample will be filtered in laboratory for samples requiring dissolved Hg analysis .
(13) Samples for dissolved mercury analysis must be filtered and preserved as soon as possible upon receipt at laboratory.
FP = Fluoropolymer (e.g., teflon)
G = Glass
P/G = plastic or glass
EPA or MCAWW = Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020.
Bottle Sources:
USGS - bottles provided by USGS, Trenton.
USGS/STL - Sample Media (and foil and bags for shipping) prepared by STL-Knoxville under contract to USGS.
STL - bottles for CLP and DESA to be purchased from STL-North Canton
Lab - Specially cleaned mercury-free bottles (and tubing) for trace mercury analysis to be provided by laboratory (STL-North Canton).

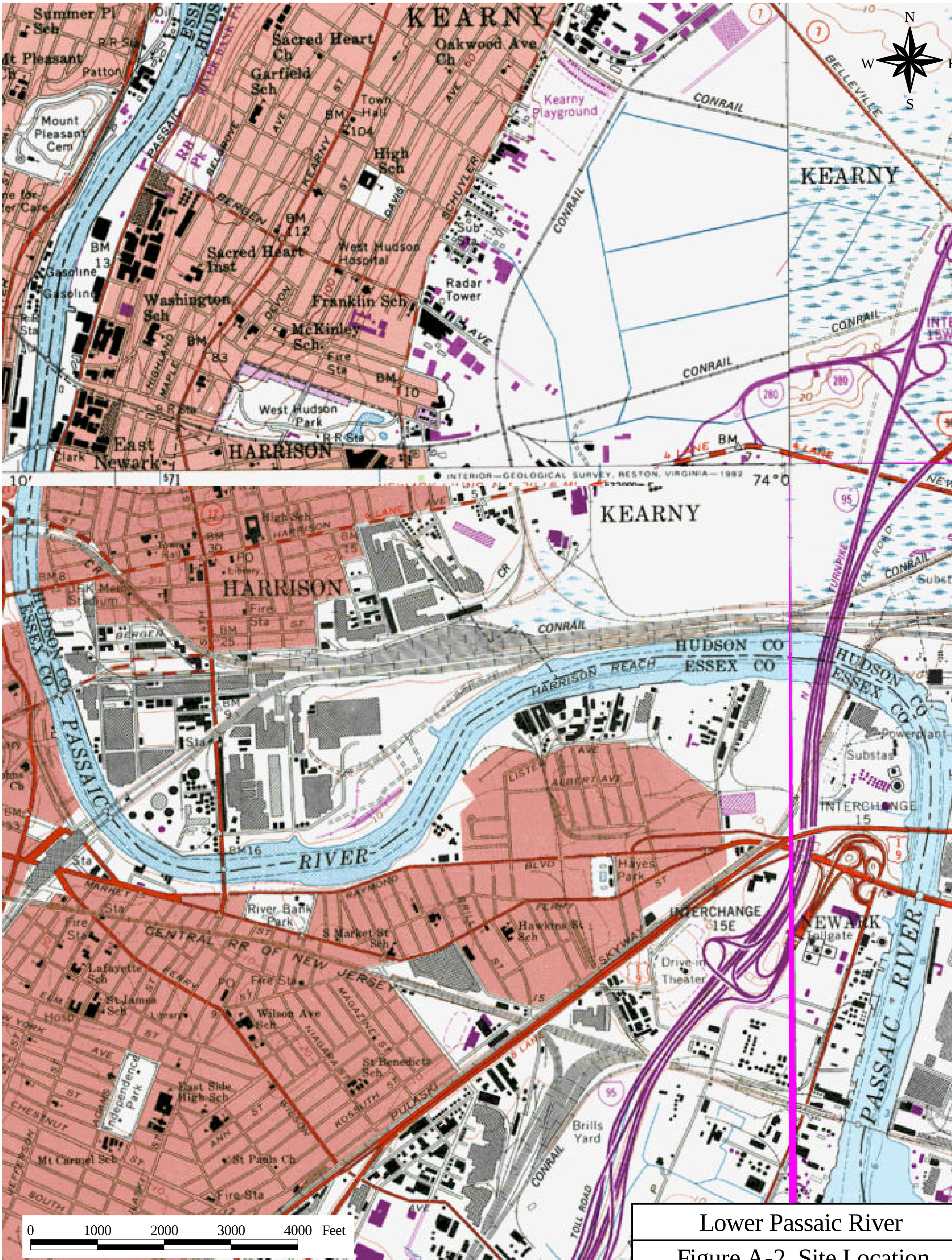
TABLE B-2
Lower Passaic River Environmental Dredging Pilot Study Quality Assurance Project Plan
FIELD SAMPLE AND QA/QC SAMPLE QUANTITY SUMMARY

	Analytical Method	Laboratory [1]	Reporting Limit Goal (units as specified)	Field Sample Quantity	Matrix Spike (MS)	MS Duplicate or Matrix Duplicate	Field Duplicate	Equipment Blank [5]	Total Analytical Samples
MATRIX/ANALYSIS									
GFF/XAD samples from TOPS [8]									
Pesticides - GFF only	SOM01.1 or OLM04.3-Pest	CLP Non-RAS	50 ng/sample (each DDX)[2]	19	1	1	0 [3]	1	22
PCBs- Congeners - GFF only	CLP CBC01.0	CLP Non-RAS	1 ng/sample/congener [2]	19	1	1	0 [3]	1	22
PCDD/PCDFs - GFF only	CLP DLM02.0	CLP Non-RAS	5 ng 2378-TCDD/sample [2]	19	1	1	0 [3]	1	22
Pesticides - XAD only	SOM01.1 or OLM04.3-Pest	CLP Non-RAS	50 ng/sample (each DDX)[2]	8	1	1	0 [3]	1	11
PCBs- Congeners - XAD only	CLP CBC01.0	CLP Non-RAS	1 ng/sample/congener [2]	8	1	1	0 [3]	1	11
PCDD/PCDFs - XAD only	CLP DLM02.0	CLP Non-RAS	5 ng 2378-TCDD/sample [2]	8	1	1	0 [3]	1	11
Metals - Whole Water									
TAL Metals (ICP-MS, w/ Al and Fe)[6]	ICP MS (OES for Fe)	DESA	0.5 ug/L	19	1	1	1	2	24
Mercury	USEPA 1631 E	Commercial [8]	5 ng/L	19	2	2	1	3	27
Metals - Dissolved (Filtered)									
TAL Metals (field filtered)[6]	ICP MS (OES for Fe)	DESA	0.5 ug/L	8	1	1	1	1	12
Mercury (lab filtered)	EPA 1631 E	Commercial [8]	0.5 ng/L	8	1	1	1	2 [11]	12
Water Samples - ISCO sampler									
Particulate Organic Carbon [7]	DESA SOP C-88	USGS/DESA [10]	100 mg/kg (particulate)	244	0	0 [12]	14	4	262
Dissolved Organic Carbon	DESA SOP C-83	USGS/DESA [10]	1 mg/L	136 [4, 9]	0	0 [12]	7	4	147
Total Suspended Solids	DESA SOP C-33	DESA	4 mg/L (100 mL sample)	462	NA	NA	24	0	486
Chloride and Bromide ion	EPA 300.0	DESA	TBD (1 mg/L by 300.0)	136 [9]	0	0	7	4	147
Water Samples - L and M Boat Grabs									
Total Suspended Solids	DESA SOP C-33	DESA	4 mg/L (100 mL sample)	50	NA	NA	3	0	53

(1) Laboratory assignments are based on information available as of November 18, 2005. Not all laboratories have been finalized at this time.
(2) Reporting goals based on 200-L samples for GFF and 20 to 50 L for XAD samples; and on published and unpublished Passaic River data.
(3) No true field duplicates are collected. However, the samples collected from the two TOPS vessels prior to dredging or debris removal are essentially duplicates of each other.
(4) Dissolved organic carbon to be determined by analysis. TOC (whole water) may be determined from POC and DOC by calculation.
(5) Mercury by method 1631E uses "equipment blank" to describe a type of lab blank. For mercury the 'equipment blank' is called 'field blank' in 1631E and frequency is one per shipment.
(6) Analyte list includes TAL metals amenable to analysis by ICP-MS (includes Al and Fe). RL shown is for lead. RL for Al is 60 ug/L. Fe analysis by ICP-OES (RL = 100 ug/L). .
(7) POC analysis is on replicates of 25 mm glass fiber filters. Report both anlayses and average. Analyze third filter if replicate precision does not meet criteria.
(8) Tentatively assigned to STL, North Canton OH facility.
(9) One-half of the 284 samples filtered for POC analysis will be (tentatively) analyzed for DOC; the Br/Cl analysis will be on 8-oz another aliquot of the same filtrate.
(10) USGS/DESA = Samples to be filtered at USGS laboratory (Trenton, NJ); dissolved phase and filters to be shipped to DESA for DOC and POC analysis and Br/Cl analysis.
(11) Field samples for total and dissolved Hg will be unfiltered. 'Field Blank' shown here will be prepared at laboratory, concurrently with laboratory filtration of samples, and rinsate blank(s).
(12) SOP requires two lab-fortified blanks or analytical quality control samples analyzed with each SDG.
CLP Non-RAS = Laboratory procured through USEPA Contract Laboratory Program, non-routine analytical services SOWs for CDD/CDFs and chlorinated biphenyl congeners.
USGS = US Geological Survey laboratory
DESA = USEPA Region 2 Division of Environmental Science and Assessment Laboratory, Edison NJ

Figure A-1
Organization Chart





Lower Passaic River

Figure A-2. Site Location

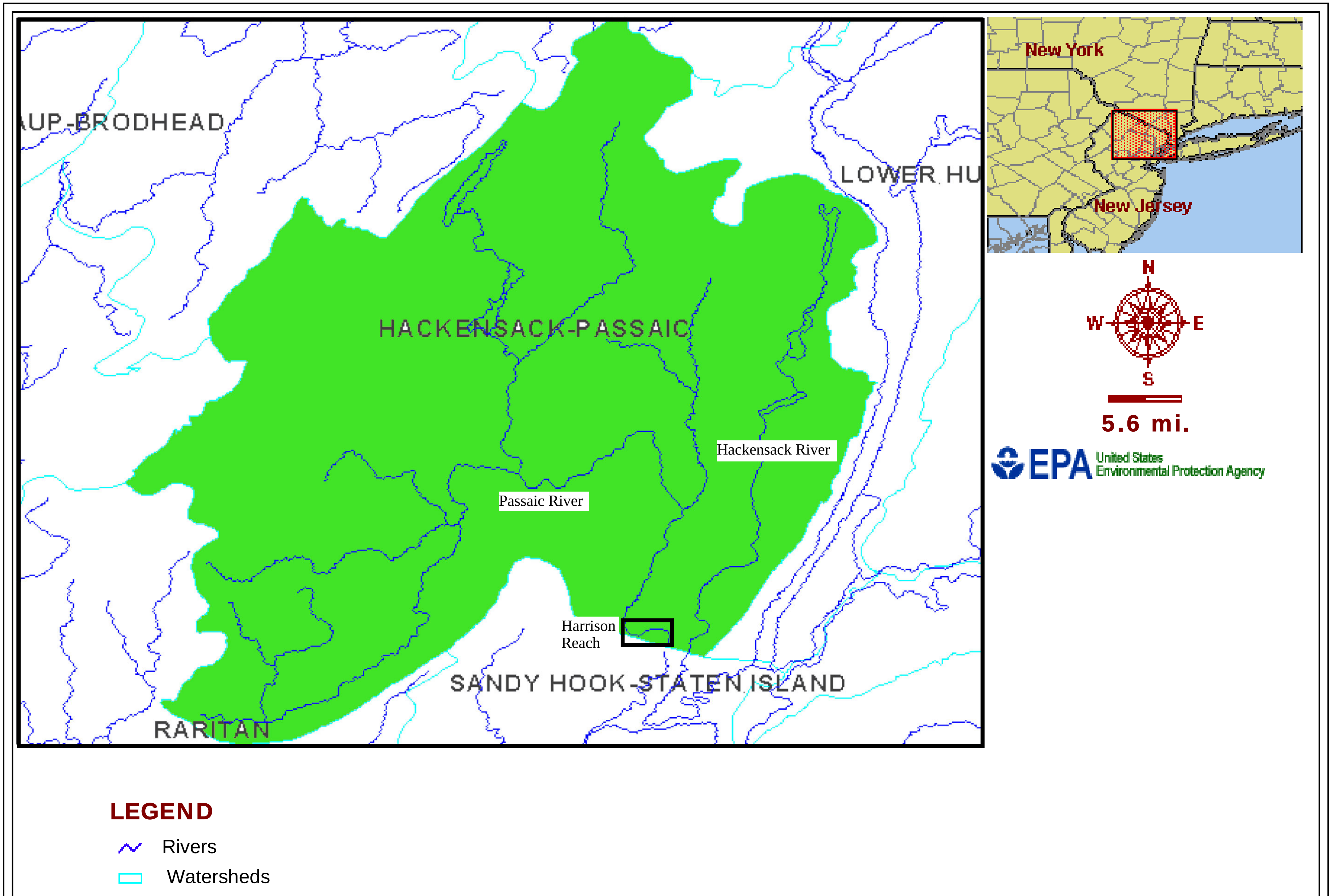
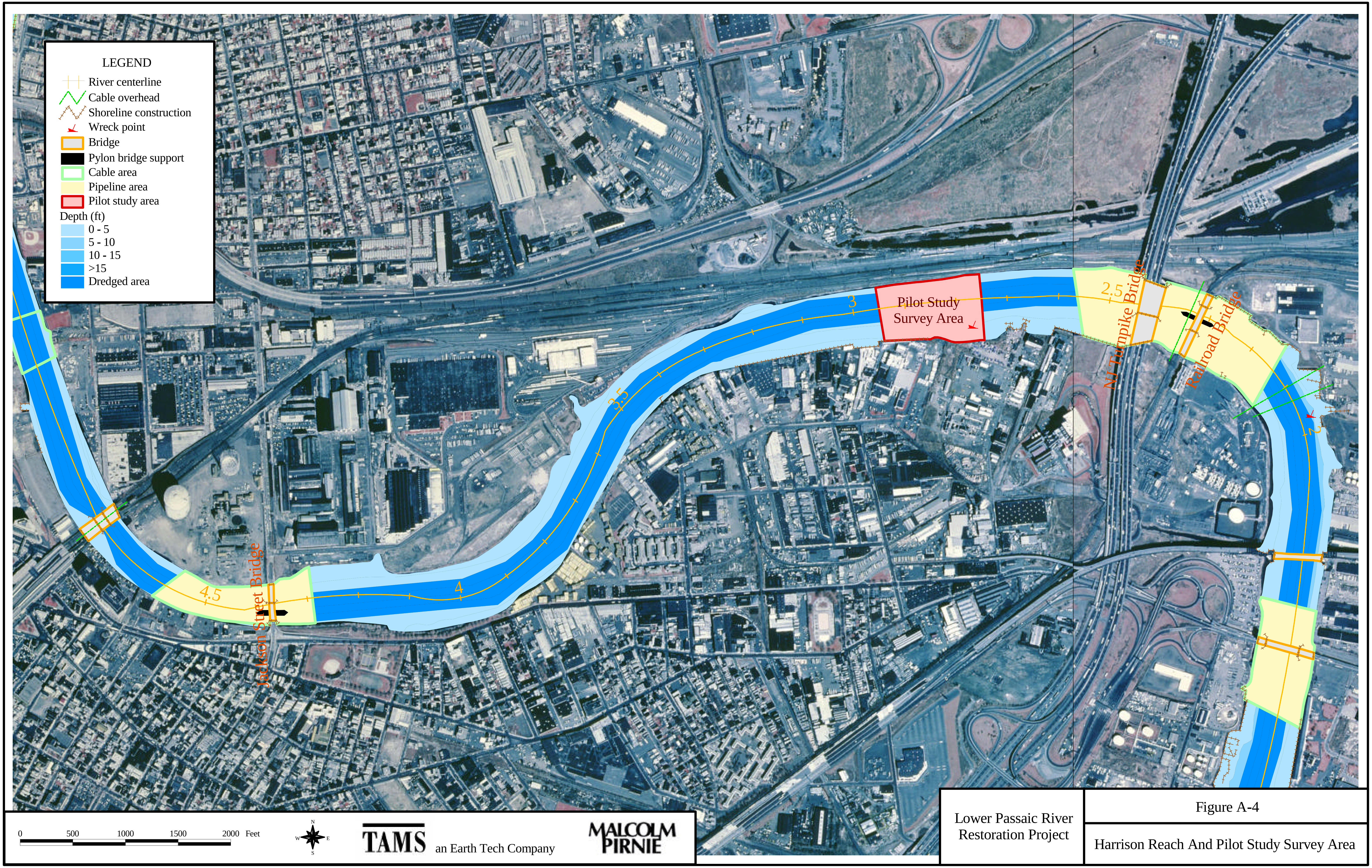


Figure A-3
Hackensack - Passaic Watershed

Reference: USEPA Envriomapper at http://www.epa.gov/enviro/html/em/index.html



Lower Passaic River Restoration Project

Figure A-4

Harrison Reach And Pilot Study Survey Area

Figure B-1: Monitoring Mooring Arrangement with Monitoring Boat Track (Plan View) Ebb Tide

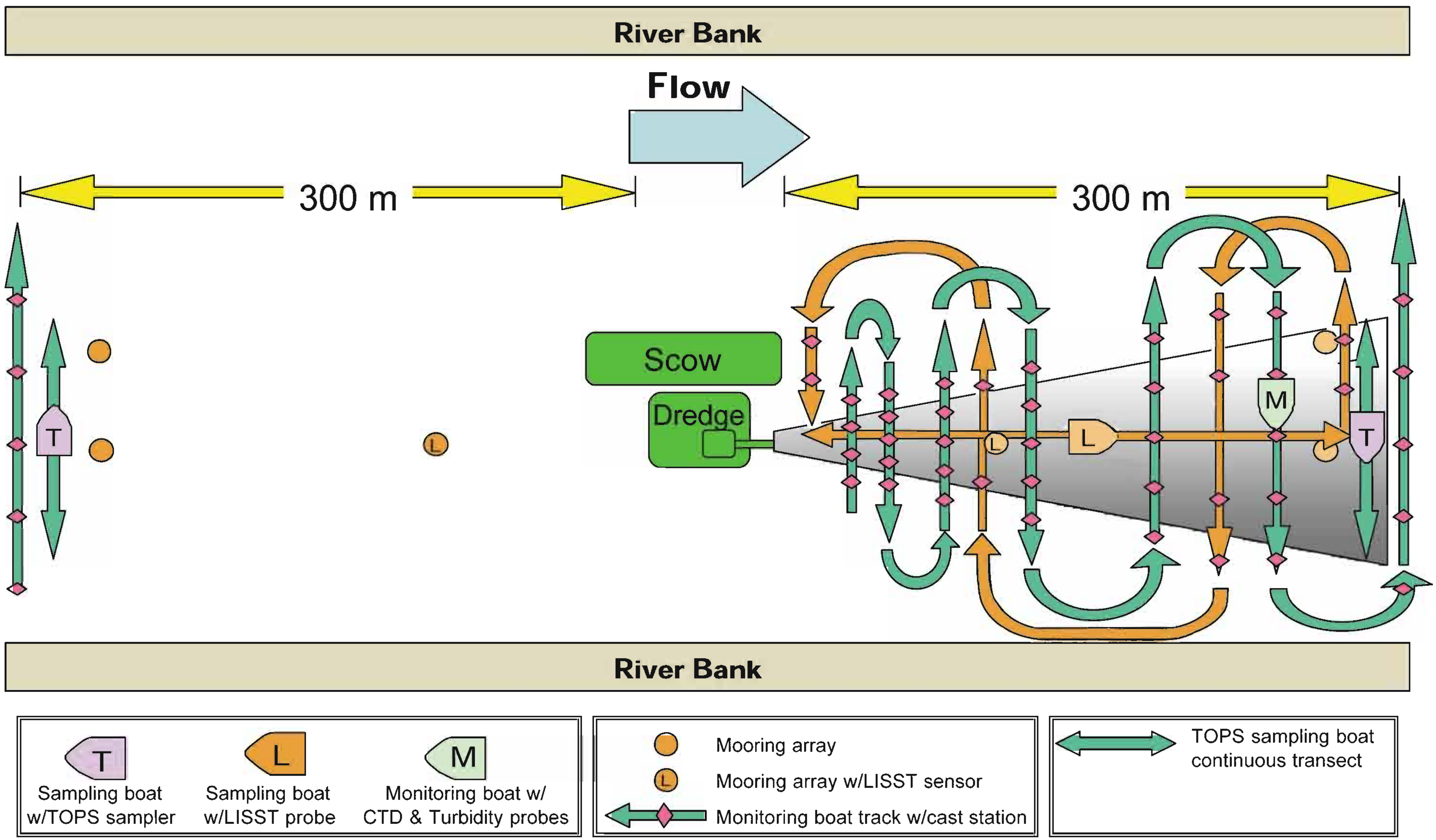


Figure B-2: Monitoring Mooring Arrangement with Monitoring Boat Track (Plan View) Flood Tide

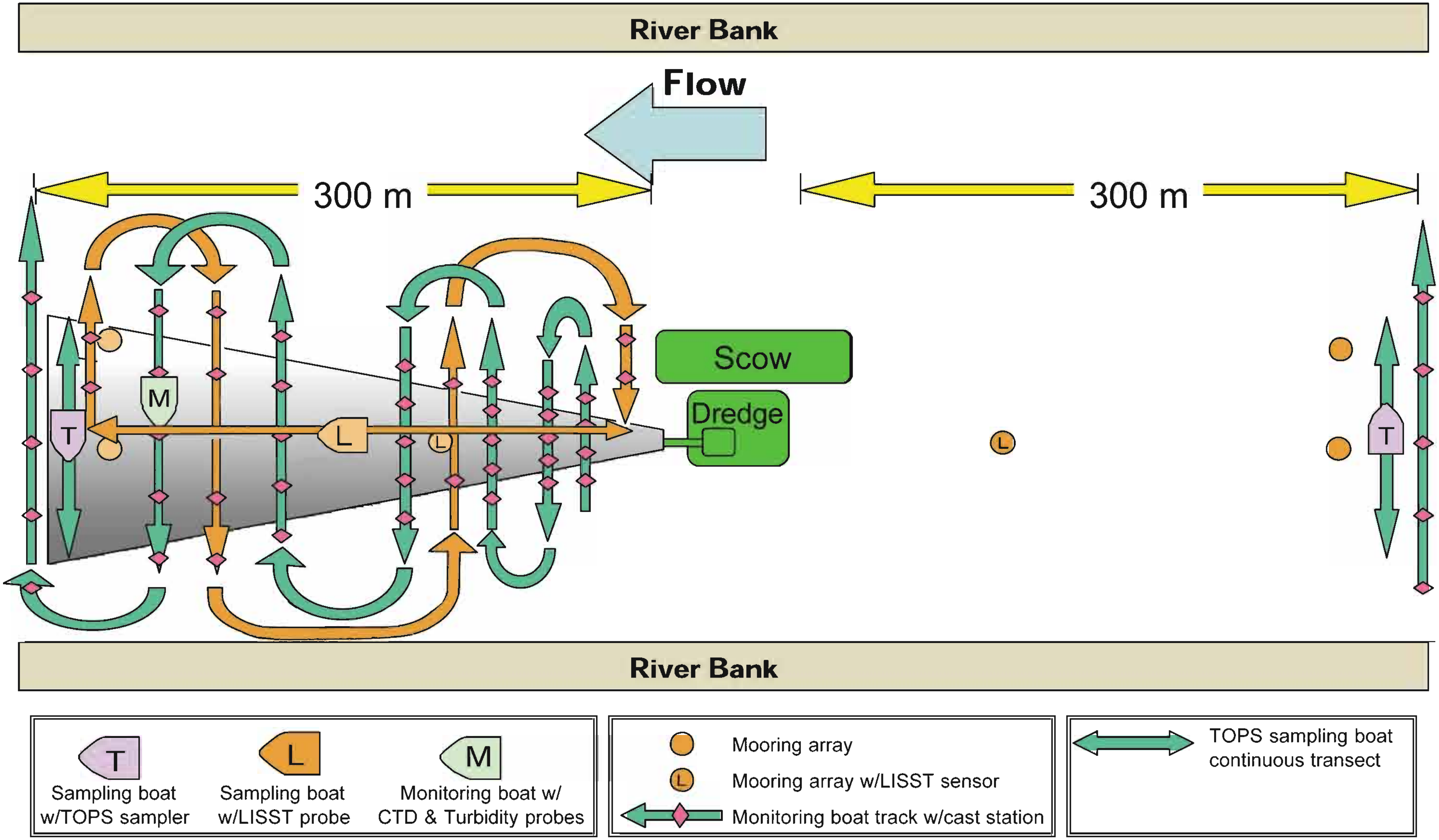


Figure B-3: Monitoring Mooring Arrangement (3D View)

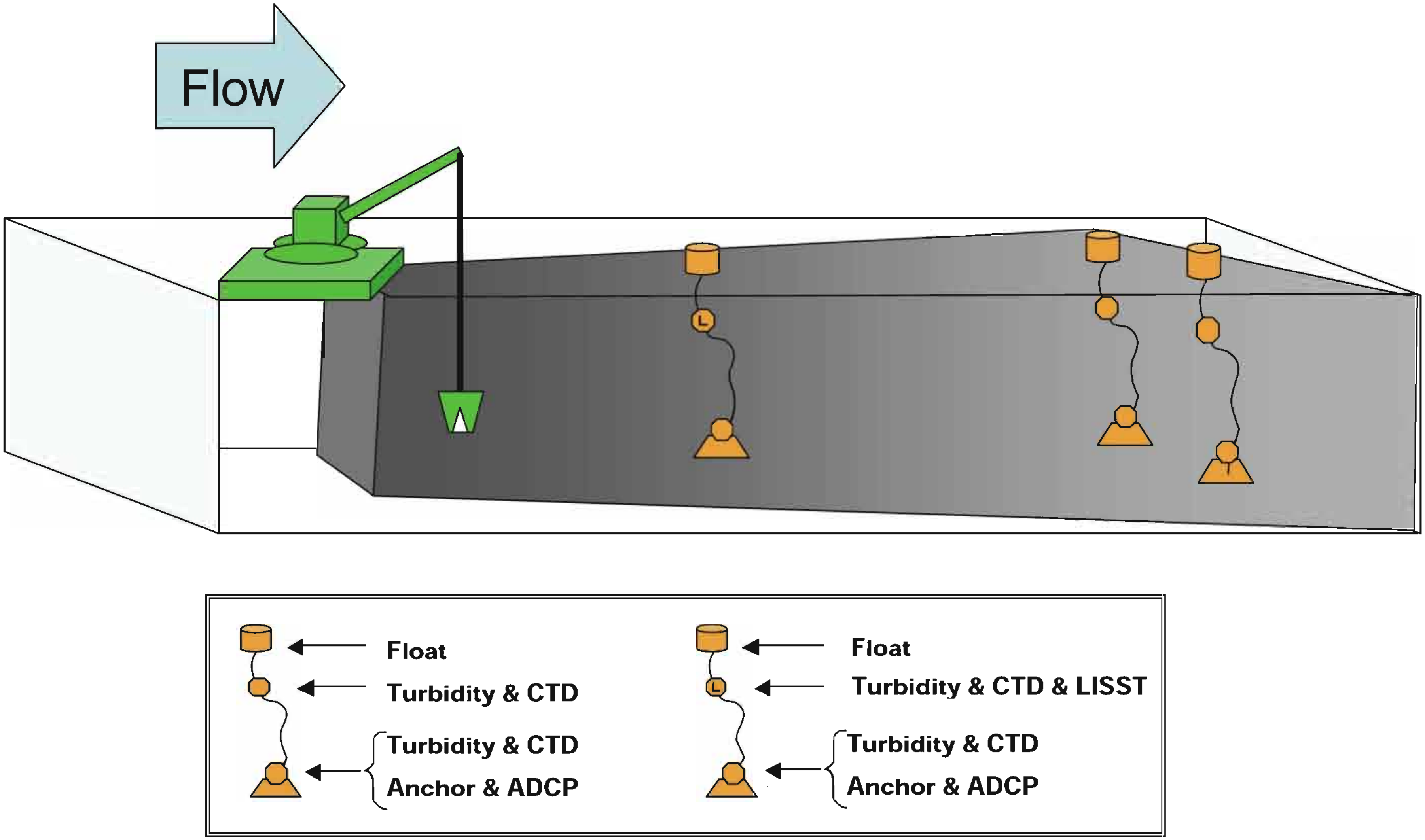
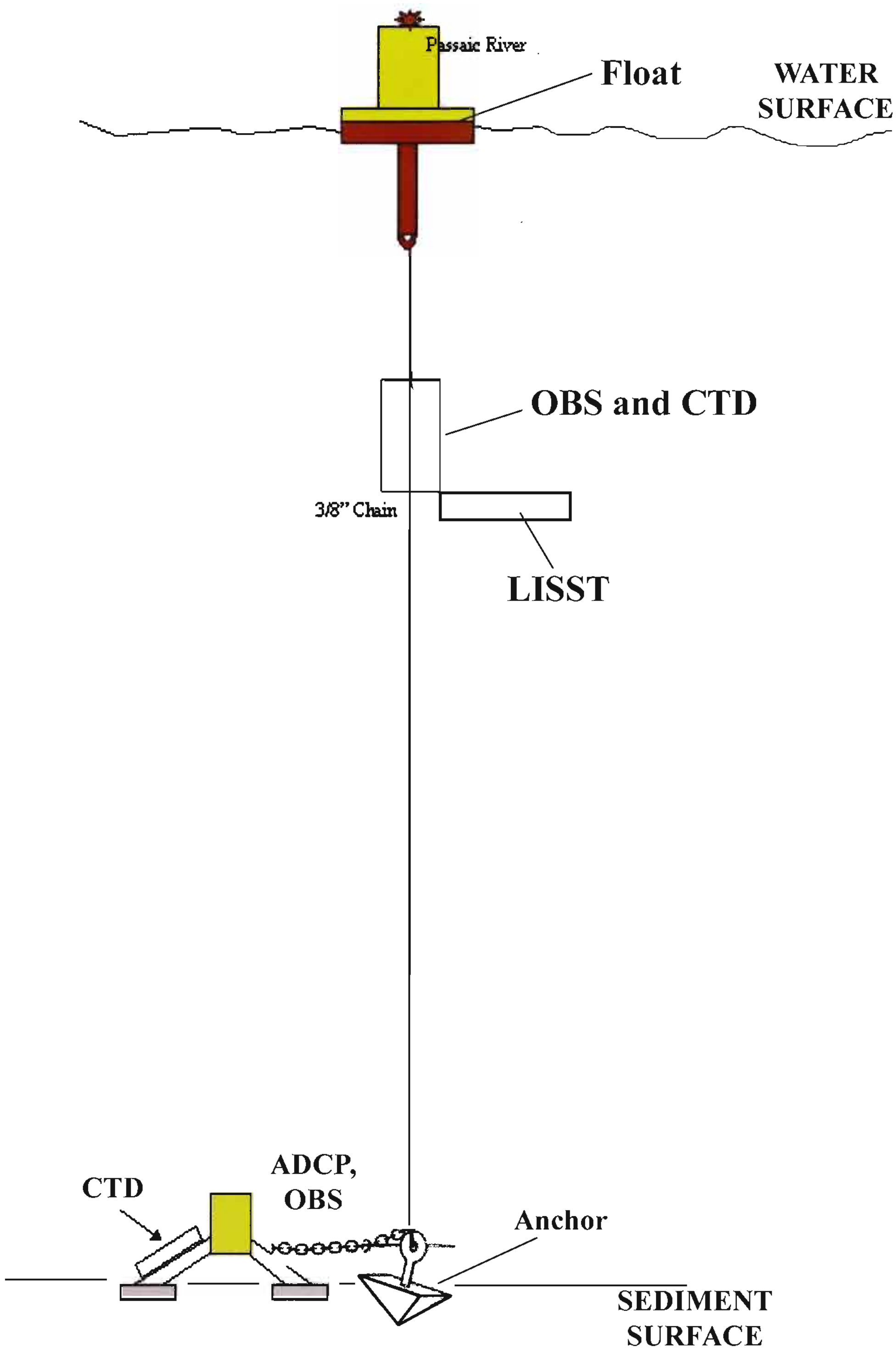
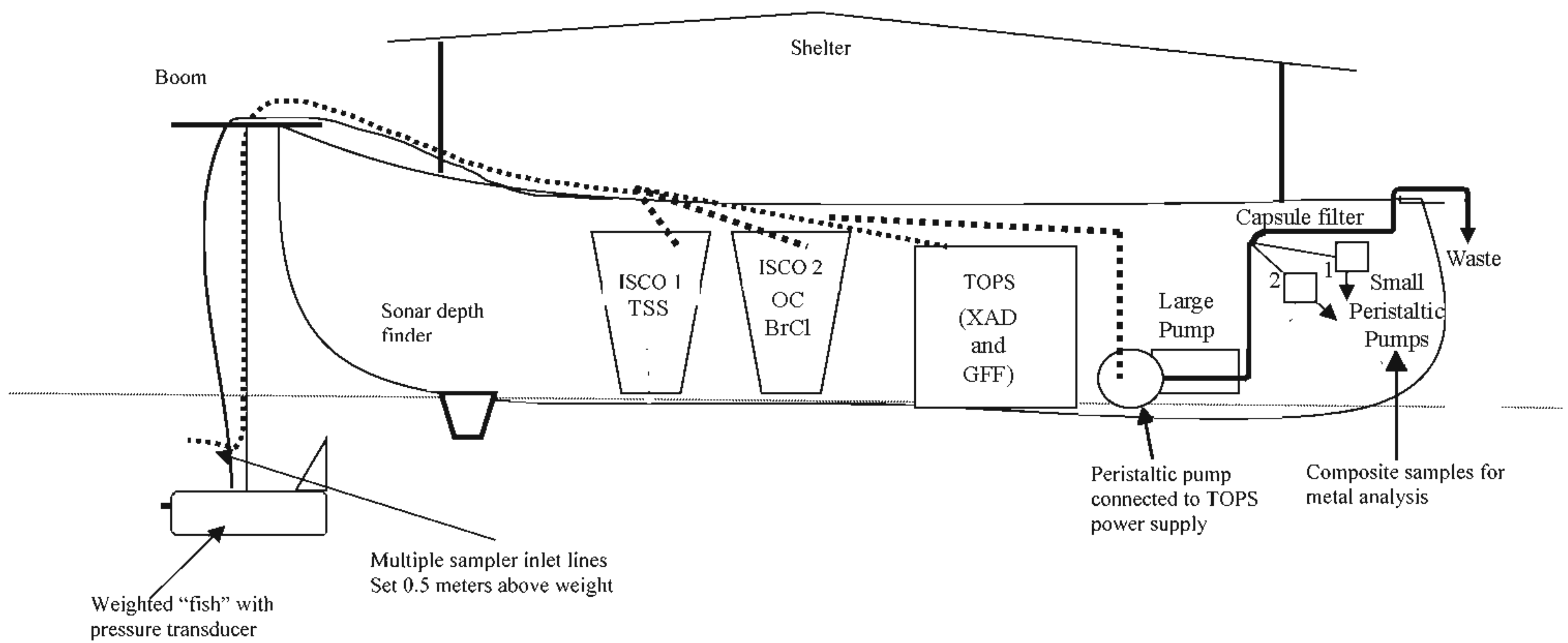


Figure B-4: Typical Mooring Setup

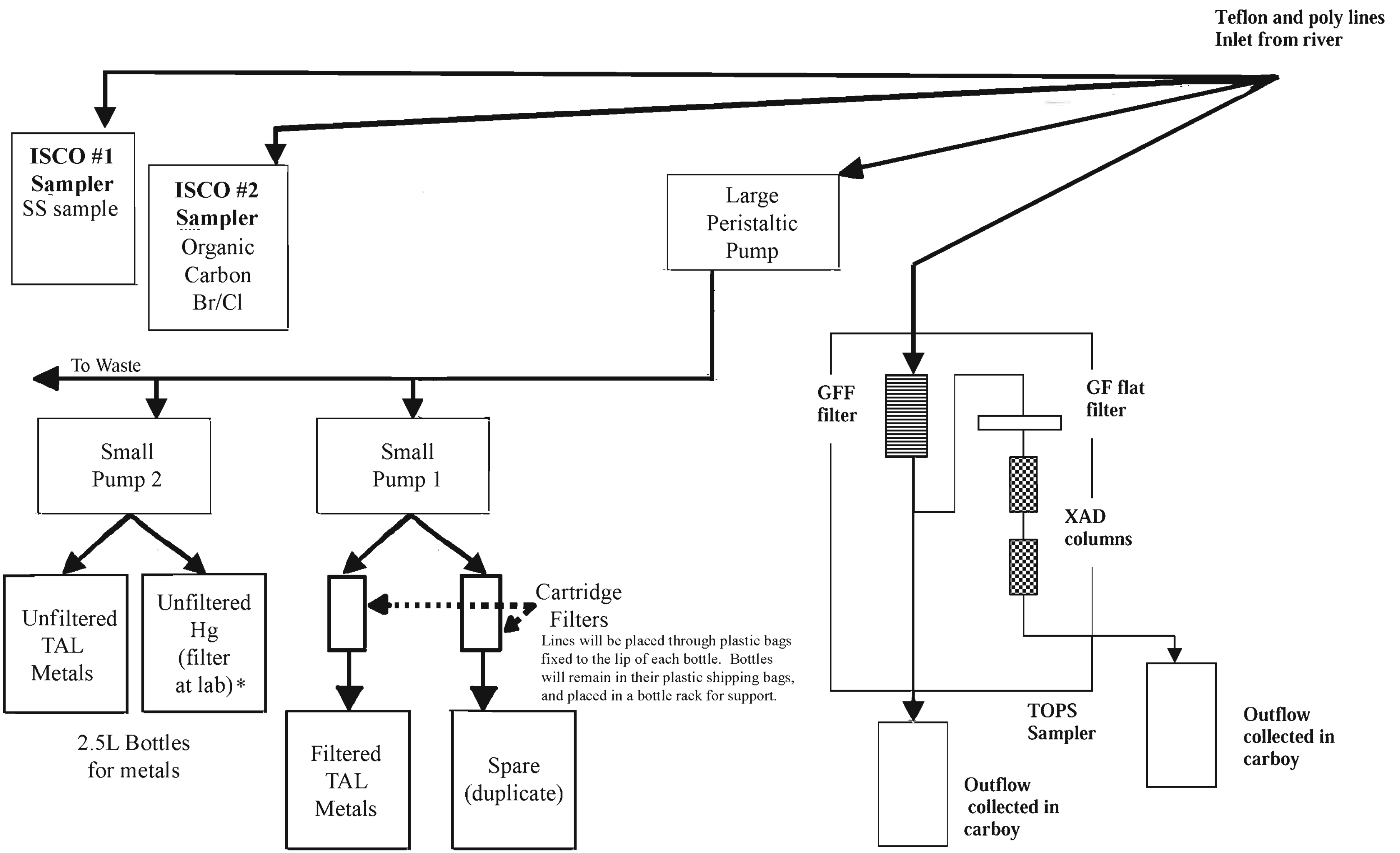




Adapted from USGS

Figure B-5. Equipment Setup for TOPS Boats

TAMS MALCOLM PIRNIE



* A single unfiltered sample for trace Hg will be submitted and analyzed for total and dissolved Hg. Sample is split at lab and one aliquot filtered immediately upon receipt.

Note: all pump tubing, filters, bottles, lab water for blanks will be provided precleaned by the laboratory. Teflon Y's used to split tubing will be precleaned following same procedure used for bottle cleaning. New Teflon tubing will be used between the pumps, filter and bottles. New pre-cleaned pump tubing will be used for each composite sample.

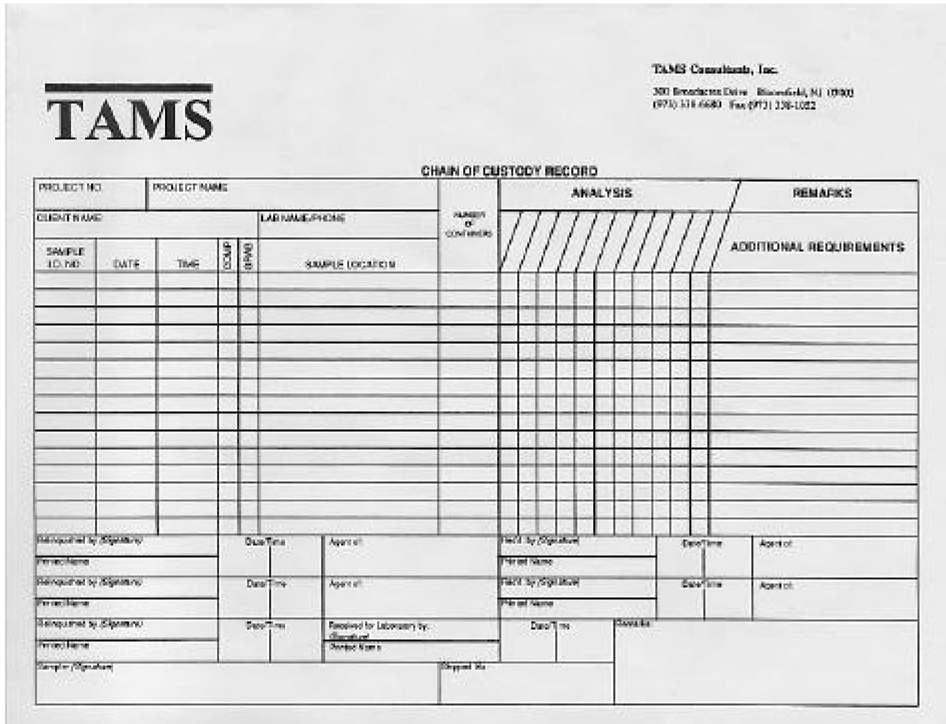
Adapted from USGS

Figure B-6. Schematic of sampling equipment in each TOPS sampling boat

TAMS MALCOLM PIRNIE

PASSAIC RIVER ENVIRONMENTAL PILOT STUDY

Example Chain-of-Custody Form



TAMS

TAMS Consultants, Inc.

CHAIN OF CUSTODY RECORD

PROJECT NO.	PROJECT NAME					NUMBER OF CONTAINERS	ANALYSIS	REMARKS
CLIENT NAME					LAB NAME/PHONE			ADDITIONAL REQUIREMENTS
SAMPLE I.D. NO.	DATE	TIME	COMP	GRAB	SAMPLE LOCATION			

Relinquished by (Signature) | Date/Time | Agent of | Rec'd. by (Signature) | Date/Time | Agent of

Printed Name | Printed Name

Relinquished by (Signature) | Date/Time | Agent of | Rec'd. by (Signature) | Date/Time | Agent of

Printed Name | Printed Name

Relinquished by (Signature) | Date/Time | Received for Laboratory by: (Signature) | Date/Time | Remarks

Printed Name | Printed Name

Sampler (Signature) | Shipped Via

FIGURE B-7: EXAMPLE CHAIN-OF-CUSTODY FORM	SCALE: NONE	EARTH TECH

APPENDIX A

Title: Water Quality Instrument Data Collection – Conductivity, Temperature and Depth.

I. Introduction

This SOP describes the equipment and methods used to collect and process *in-situ* water quality data for conductivity, temperature, and depth (CTD), the three primary parameters measured with a submersible water quality instrument. This procedure is to be followed for the Lower Passaic River Restoration Project. Any substantive modification to the procedure shall be approved in advance by the Field Team Leader(s), the Site Quality Control Officer, and/or the Project Manager.

This procedure defines general methods used to collect water quality data using electronic sensors. Specific methods and materials are detailed in the individual instrument and sensor operating manuals, and the manufacturer developed software.

II. Definitions

CTD: Submersible conductivity, temperature, and depth instrument.

III. Equipment and Supplies

The following equipment is required to collect and store water quality data:

1. CTD instrument with communication cable.
2. Laptop computer with instrument interface software, field sampling data collection application.
3. Batteries for the instrument.
4. Vessel with DGPS navigation system.
5. Small weight (if instrument is not in a cage) and flotation for instrument to maintain the instrument in a vertical orientation against light currents.
6. Line (⅜" - ¾" diameter) to deploy instrument (*e.g.*, nylon, polypropylene; braided construction recommended).
7. Personal safety gear- , including personal flotation devices (PFDs), waterproof outer wear, steel toed boots (waterproof if rough seas or weather), and mobile phone.
8. Calibration materials
   - Conductivity/Salinity- Conductivity solutions (two concentrations recommended).
   - Temperature- NIST thermometer.
   - Depth- Measuring tape and weight

6. After a successful profile, enter prompted information into the field application:
   - Date
   - Time of water column profile
   - Actual coordinates of the sample location
   - Water depth (feet)
   - Instrument Serial Number
   - Sensor data collected
   - Observations

7. At the end of each day, an electronic copy (disk) of the field application that includes the information recorded for each profile collected that day will be created as a back up of that day's project information. A copy of the signed field log form will be maintained by the field team leader.

Title: Physical Oceanographic Data Collection with Acoustic Doppler Current Profiler (ADCP).

I. Introduction

This SOP describes equipment and methods used to collect and process *in-situ* physical oceanographic data for current speed and direction. This procedure is to be followed for the Lower Passaic River Restoration Project. Any substantive modification to the procedure shall be approved in advance by the Field Team Leader(s), the Site Quality Officer and/or the Project Manager.

This procedure defines general methods used to collect hydrodynamic data using electronic sensors. Specific methods and materials are detailed in the individual instrument and sensor operating manuals, and the manufacturer developed software.

II. Definitions

ADCP: Acoustic Doppler Current Profiler instrument.

SCUBA: Self Contained Underwater Breathing Apparatus

III. Equipment and Supplies

The following equipment is required to collect and store water quality data:

1. ADCP instrument with communication cable.
2. Laptop computer with instrument interface software, field sampling data collection application, and backup power supply.
3. Battery with adequate charge state for the instrument deployment plan.
4. Vessel with DGPS navigation system.
5. Mooring, site and project specific design;
    - Water column mooring. Primary weight (>300 lbs, if instrument is not in a trawl proof mooring) with two eyes (smooth); one for main mooring line and one for ground line. Secondary weight (>30 lbs, if instrument is not in a trawl proof mooring) with one eye to attach ground line.
    - Trawl proof mooring – typically pyramid shaped mooring that allows trawl nets to pass over without moving or fouling mooring. Includes small float and acoustic release to retrieve or the use of SCUBA divers to locate and attach recovery line.

6. Line (½" - ¾" diameter) to deploy instrument (*e.g.*, nylon, polypropylene; braided construction recommended). ~20 ft for instrument and floats, and 100' for ground line.
7. Floatation to maintain instrument in vertical position.
8. Acoustic pinger to assist in recovery if instrument moves during deployment.
9. Misc. field supplies (*e.g.*, electrical tape, cable ties, anti-chafe materials, knife, hand tools).
10. Personal safety gear, including personal flotation devices (PFDs), waterproof outer wear, steel toed boots (waterproof if rough seas or weather), and mobile phone.
11. Manufacturer's instructions for calibration and deployment programming.
12. Oceanographer's data requirements and approval of deployment plan
13. Deployment log, indelible markers/pens (*e.g.*, Sharpies™).

IV. Guidelines

Deployment

1. ADCP instruments must be calibrated at least as frequently as the manufacturer's recommendation. Typical service interval is yearly, and is primarily for water tight integrity maintenance.

2. Using manufacturer's software, follow manufacturer's instructions to test and calibrate instrument prior to programming. When successfully prepared, program instrument as described in the Work Plan or according to written instructions from physical oceanographic task leader. Have second staff member confirm settings against plan.

3. Prepare main mooring by:

   - Attaching all equipment (primary weight, instrument, floatation, pinger, ground line, ground line weight). The detailed mooring design is Work Plan and location specific.

   -or-

   - Preparing trawl proof mooring as designed by manufacturer.

4. Either mooring may or may not include an acoustic release. If an acoustic release is included, use fresh batteries and test the acoustic release prior to deployment.

5. Position ship at target location.

6. Deploy meter in accordance with directions given by the captain. Extreme caution, good planning, and excellent communication are necessary for safe and successful deployments. The combination of heavy mass on a moving platform with delicate instruments creates situations with high levels of risk for staff and equipment.

7. Record position of deployment with as much accuracy as possible (*i.e.*, stay on location if possible and average as many DGPS location fixes as possible (>20 is preferred).

8. Upon completion of deployment, all ancillary data (*e.g.* date, time, position) is entered into the field application. The field application prompts the user for the required information and also automatically uploads daily weather and tidal conditions from the NOAA website. Blank field log sheets to record information manually will be provided in case difficulties with data entry into the field computer are encountered.

Recovery

Weight/ floatation mooring without acoustic release

1. Position vessel near mooring.
2. Following captain's instructions, drag grappling hook for ground line.
3. When line is tight and mooring is hooked, slow vessel and follow captain's instructions, recovering the mooring slowly as safely and gently as possible to the vessel deck.

Weight/floatation with acoustic release

1. Position vessel within ~20 meters of mooring. Engage as many crew members as possible to scan water for visual contact with released float.
2. Activate acoustic release, following manufacturer's instructions.
3. When float is sighted, follow captain's instructions for recovery of mooring.

Diver located mooring

Position vessel near mooring:

1. Deploy marking float with weight as close to mooring coordinates as possible.
2. Divers descend along marking line, and perform systematic search for mooring.
3. Divers locate mooring, attach recovery line, and return to surface.

4. Follow captain's instructions for recovery.

As with deployment, heavy weight and delicate instruments create situations of above average risk during recovery operations.

5. After a successful recovery, download instrument data to field computer/field application.
6. Enter prompted information into the field application:

   - Date
   - Time of meter recovery
   - Actual coordinates of the recovery location
   - Water depth (feet)
   - Instrument Serial Number
   - Sensor data collected
   - Observations

7. At the end of each day, an electronic copy (disk) of the field application that includes the information recorded for each set of physical oceanographic data is to be collected. A copy of the signed field log form will be maintained by the field team leader.

Title: Water Quality Instrument Data Collection – Turbidity

I. Introduction

This SOP describes the equipment and methods to collect and process *in-situ* water quality data for turbidity. This procedure is to be followed for the Lower Passaic River Restoration Project. Any substantive modification to the procedure shall be approved in advance by the Field Team Leader(s), the Site Quality Control Officer, and/or the Project Manager.

This procedure defines general methods used to collect water quality data using electronic sensors. Specific methods and materials are detailed in the individual instrument and sensor operating manuals, and the manufacturer developed software.

II. Definitions

CTD: Submersible conductivity, temperature, and depth instrument.

NTU: Nephelometric Turbidity Units

RTU: Relative Turbidity Units

III. Equipment and Supplies

The following equipment is required to collect and store water quality data:

1. CTD instrument with communication cable.
2. Optical (Turbidity) sensor.
3. Laptop computer with instrument interface software, field sampling data collection application.
4. Batteries for the instrument.
5. Vessel with DGPS navigation system.
6. Small weight (if instrument is not in a cage) and flotation for instrument to maintain the instrument in a vertical orientation against light currents.
7. Line (⅜"-¾" diameter) to deploy instrument (*e.g.*, nylon, polypropylene; braided construction recommended).
8. Personal safety gear, including personal flotation devices (PFDs), waterproof outer. wear, steel toed boots (waterproof if rough seas or weather), and mobile phone.
9. Calibration materials: turbidity solutions (minimum of two).
10. Station logs, indelible markers/pens (*e.g.*, Sharpies™).

IV. Guidelines

1. The turbidity sensor is one of two types of optical sensors and measures either 'backscatter' by emitting a light and measuring reflected return, or 'transmissivity' which emits a light and measures return over a measured distance (typically 10 cm). Data is reported in either NTUs or RTUs. The sensors are relatively stable, and must be calibrated yearly, at a minimum. However, they are not difficult to calibrate and it is recommended they be calibrated prior to each survey, or after any signs of degradation in data quality.
   - Visual signs of sensor degradation are discolored optical surfaces or reduction in light emittance.
   - Signs of degradation in the data quality are slow response time, limited response magnitude, high noise in data, and unmatched upcast and downcast data curves.

2. Prior to the survey, calibrate the sensor as per manufacturer specifications in a minimum of two calibration solutions. Typically, a clear (0 NTU) solution is used, and a second solution near the average turbidity level at the site is used. Note pre-calibration values and instrument voltage settings post-calibration in the field application or instrument logbook.

3. Deploy CTD instrument (with turbidity sensor) as described in the SOP #PR-27: Water Quality Instrument Data Collection – Conductivity, Temperature and Depth.

4. Turbidity data collected from the CTD is recorded in the field computer. The instrument is either directly connected via electronic cable to the computer, or data is stored in the instrument, and transferred periodically during the survey. Upon completion of sampling at one location, all ancillary data (*e.g.*, date, time, position) is entered into the field application. The field application prompts the user for the required information and also automatically uploads daily weather and tidal conditions from the NOAA website. Blank field log sheets to record information manually will be provided in case difficulties with data entry into the field computer are encountered.

5. Turbidity profile data should be reviewed as often as is practical to assure results are within expected ranges and the instrument is operating properly. Graphical plots or spreadsheet average/minimum/maximum review are suitable methods. For graphical plots, comparison of down-cast and up-cast data plots is useful. If data is suspect, the discrepancy is noted in the field application, and the meter should be re-calibrated as soon as possible.

6. After a successful profile, enter prompted information into the field application:

- Date.
- Time of water column profile.
- Actual coordinates of the sample location.
- Water depth (feet).
- Instrument Serial Number.
- Sensor data collected.
- Observations.

7. At the end of each day, an electronic copy (disk) of the field application that includes the information recorded for each profile collected that day will be created as a back up of that day's project information. A copy of the signed field log form will be maintained by the field team leader.

LISST-100

Particle Size Analyzer

User's Manual

Version 4.2

SEQUOIA

15317 NE 90th Street
Redmond, WA 98052
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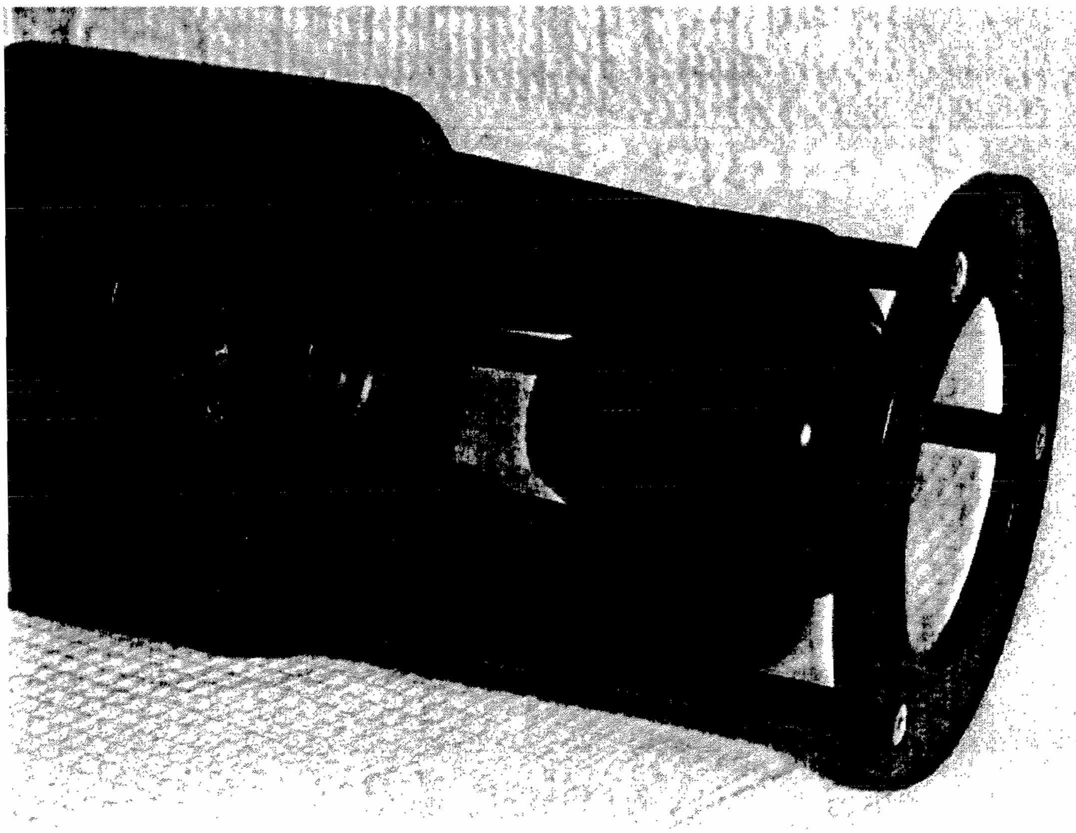


SEQUOIA

Welcome to the LISST-100 Particle Size Analyzer

Using this manual

This manual is divided into two sections.

Section One contains an introduction to the LISST- 100 instrument and the principles of its operation.

Section Two provides a detailed set of instructions for using and caring for the instrument.

Instrument specifications

For a listing of instrument-specific constants, as used in the software for data processing, refer to Appendix A.

Technical assistance

For technical assistance please contact your local Distributor or a Sequoia Technical Service Representative listed below. Please be sure to include the instrument serial number with any correspondence.

Factory Technical Service Representatives

Chuck Pottsmith
pottsmith@sequoiasci.com; (425) 867-2464 ext.107

Doug Keir
keir@sequoiasci.com; (425) 867-2464 ext.104

Table of Contents

Section 1: Introduction to the LISST-100

Introduction

Principle of Operation

The product name LISST is derived from the term that describes its operation: Laser In-Situ Scattering and Transmissometry. LISST is a Trademark of Sequoia Scientific, Inc.

The LISST-100 instrument uses the technique of laser diffraction to obtain *particle size-distribution* (PSD), also called *volume distribution* in this manual. This section describes the principle of operation of your instrument.

The laser diffraction method for sizing particles was invented in the 1970's and rapidly became the most widely used optical method for determining size distribution for the simple reason that for laser diffraction, the composition or refractive index of the particles is not important. This method determines size distribution of an ensemble of particles, as opposed to counting type devices that size one particle at a time. The reason that laser diffraction is unaffected by composition of particles is that the scattering of laser light is observed at multiple, *small* forward angles. At these small angles, light scattering is determined almost entirely by light *diffracted* by the particle. The light *transmitted* through the particle makes only a weak contribution to the measured scattering. Since the light transmitted through the particle would *experience* the composition of the particle, i.e. its refractive index, and since it makes only a weak contribution

A collimated laser beam enters water, light is scattered by particles and sensed by a multi-ring detector behind a receiving lens. A photodiode placed behind a centered hole measures optical transmission

to the observed scattering, the method of laser diffraction is mostly *independent* of particle composition. In the aquatic sciences, particle refractive index is poorly known. Thus, except for shape effects, laser diffraction offers an excellent method for size-distribution estimation. Shape effects are a matter of continuing research and no definite generalized guidelines are available to account for them *by any method*. Consequently, laser diffraction methods deliver the equivalent-spheres size distribution. By calibrating for concentration with ISO standard natural particles, shape effects are empirically included.

Continued on the next page...

The LISST-100 records the scattering intensity over a range of small angles using a specially constructed multi-ring detector shown in the graphic above. This measurement is also known to optical scientists as the *volume scattering function* and can be used as such for studies of underwater image propagation and beam spread. A growing number of publications of measure-ments using the LISST-100 instrument is available on our website: http:www.sequoiasci.com.

The conversion of the multi-angle scattering to size distribution involves a mathematical *inversion*. In this step, a size-distribution is found that would produce multi-angle scattering that fits the observation. There exists a vast literature on the subject of this inversion. For the stout-hearted user interested in learning all about it, we recommend the most germane study of the subject:

> "Optimal scaling of the inverse Fraunhofer diffraction particle sizing problem: The linear system produced by quadrature," by E. D. Hirleman, *Particle Characterization*, 4, 128-133, 1987.

The main conclusions are that the information content of the data is most stably retrieved by use of ring-type detectors, with each ring measuring the scattering over a sub-range of angles, and that the resolution is limited to relatively few size classes depending on the noise in the data. For example, for the dynamic range in scattering angles of 200:1 that is built into the LISST systems, typically only 12-14 truly *independent* size classes can be resolved.

Simpler descriptions of principles and practice are offered in a separate study by Agrawal et al., 1991[1]. A more up to date description of the technology and its application is provided by Agrawal & Pottsmith, 2000[2].

We explain the *inversion* process as follows: Let $\underline{E}$ be the 32-element data vector that contains the angular scattering energy sensed by the 32 silicon rings. It is related to the volume distribution[3] via

$$\underline{E} = \underline{K}\, \underline{N}_V$$

where $\underline{K}$ is the scattering property kernel matrix and $\underline{N}_V$ is the volume distribution.

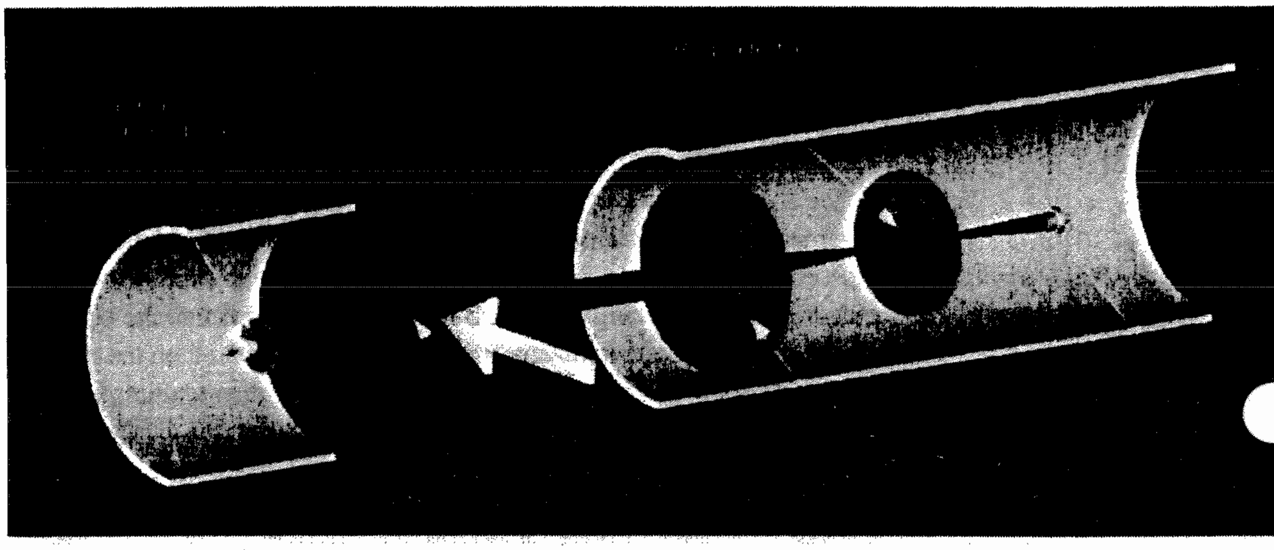
[1] Agrawal, Y.C., I.N. McCave, and J.B. Riley, 1991: "Laser diffraction size analysis," in *Principles, methods and applications of particle size analysis*, J.P.M. Syvitski, editor, Cambridge University Press, pp. 119-128.

[2] Agrawal, Y. C. and H. C. Pottsmith, 2000: Instruments for Particle Size and Settling Velocity Observations in Sediment Transport, Marine Geology, 168, pp 89-114.

The volume distribution is obtained by inverting the above equation. To test whether the estimated solution is a good one, one may compute the prediction of a scattered energy estimate $\underline{E}_e$ as

$$\underline{E}_e = \underline{K}\,\underline{N}_e$$

If the measurements $\underline{E}$ are consistent with $\underline{E}_e$, the estimates of $\underline{N}_e$ can be considered good. Included with the LISST-100 instrument, we have provided software that obtains the inverted volume distribution $\underline{N}_V$. We have also provided the matrix $\underline{K}$ for your instrument (in ASCII and MATLAB® formats) in order for you to test your results.



[3]The volume distribution is related to the number distribution via $N_V = a^3\, n(a)$. It represents the volume concentration of particles of particles in the size range a to $a + da$. The results from the LISST instruments provide the volume concentration of particles in each of 32 size classes. The size classes are log-spaced from the range 1.25-250 microns (Type-B instruments), 2.5-500 (Type-C), or 7.5-1500 microns (Type-D, LISST-FLOC). See Appendix B for further details.

The LISST-100 instrument is a laser diffraction device. It consists of optics for producing a collimated laser beam, a specially constructed detector array, electronics for signal pre-amplification and processing, a data storage and scheduling computer, and a battery system.

The instrument is capable of autonomous operation. For such operation, software is provided to program the instrument for a specific sampling schedule. The primary measurements delivered by the LISST-100 are the small-angle scattering properties of particles in water, the laser optical transmissivity, pressure, and temperature. An auxiliary Analog port is available for recording data from an external input.

After recovery of the instrument, small-angle scattering data are off-loaded from the instrument and subsequently *inverted* mathematically on a PC to produce the particle size distribution. The inversion function is included in the software.

The principal measurement—angular scattering distribution— is obtained over 32 ring-detectors whose radii increase logarithmically from 102 to 20,000 microns. The detector is placed in the focal plane of the receiving lens. The rings cover an angular range from 0.0017 to 0.34 radians (Type-B), 0.00085-0.17 (Type-C), or 0.00028-0.053 radians (Type-D, or LISST-FLOC) . This angular range corresponds, respectively, to size rangesL 1.2 to 250 microns (Type-B), 2.5-500 (Type-C) and 7.5-1500 microns (Type-D, or LISST-FLOC).

General layout

This self-contained instrument consists of the following parts: a solid-state laser operating at 670nm wavelength and fiber-optically connected to a laser beam collimating system, a beam manipulation and orienting system, a scattered-light receiving lens, the specially designed 32-ring detector, preamplifier electronics, a ring-selecting multiplexer circuitry, and a data logger.

Depth and Temperature

In addition to measuring the particle size and concentration, the LISST-100 also has built-in Depth and Temperature sensors. The Depth sensor is a 500 psi strain gauge sensor. The stainless steel fitting on the Optics endcap is used for testing this sensor. When not used for testing, this fitting helps keep contamination out of the pressure sensor. The temperature is measured using a high precision thermistor imbedded into the Optics end cap. Both values are stored automatically in the standard-format LISST-100 datafile.

External I/O Ports

The LISST-100 is also equipped with an external ANALOG IN port for recording the output of one other analog instrument. Additionally, 2 DIGITAL I/O ports are provided for communicating with up to two instruments. The I/O ports can be used for synchronization of the LISST-100 with two other instruments; e.g., the LISST-100 may provide a timing pulse to a CTD, or it may provide a 'start sample' pulse to another LISST-100. The same ports can be used to receive similar commands. Contact Sequoia Scientific for more information on interfacing the LISST-100 with other instruments. These ports are available on the 6-pin underwater connector on the Connector Endcap. Appendix C shows the connector pinouts. Software provided with the instrument accepts data acquisition commands.

Battery Life

The primary power source for the LISST-100 is a custom alkaline D-cell battery-pack. The pack outputs +18, +15 and –18 volts. It has a room-temperature capacity of 14 A-hr on each of the +18 and –18 sides. When the instrument is in stand-by mode, (i.e., when the program is not executing instructions other than SLEEP and when the serial communication cable is disconnected) the current drain is 2 mA. If used as advised, the battery provided in the instrument has a capacity to power the instrument for up to a year. A data-protecting backup battery (one-year life) can extend *in-situ* deployment beyond a year. For profiling applications, where the instrument is sampling continuously, the battery pack has about 60 hours of “On” time. Powering down the instrument between samples can greatly extend the deployment times. An EXCEL spreadsheet is included with the software that can estimate the battery consumption

For laboratory or tethered usage, a connector is provided on the endcap to draw power from an external source. The voltages required from the external power supply are +15 and –15 volts. Cables up to 50 meters can be provided to supply external power or for communication with the instrument. This can allow Real-Time observation of the Size Distributions.

When not in use, use the Low Power Sleep mode of the instrument to reduce battery drain to about 10µA. This can be done from the provided software.

Auxiliary Equipment

The instrument is shipped pre-aligned and tested. A chamber is provided for obtaining measurements of background scattered light from optical surfaces. This background is subtracted from actual particle scattering measurements to obtain the true particulate scattering. Additionally, small tools used to open endcaps, spare batteries, and communication cable are supplied so that a user need only provide a PC.

Software is provided to communicate with the instrument, schedule an experiment, offload the data, and invert the measurements to obtain particle size distribution and volume concentration. For laboratory use or for monitoring the progress of an experiment, the software can be used for real-time processing.

For laboratory testing an optional Small Volume Full Path Mixing Chamber is available. This chamber mounts to the optics of the LISST-100 and seals around the windows of the instrument. The sample can then be added to the chamber and the built-in mixer will keep the sample mixed with the measurements are being made. The picture below shows the chamber being installed onto the LISST-100.

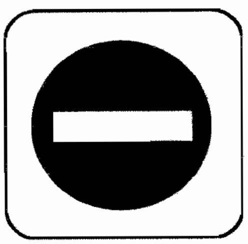
Section 2: Operation

Section Organization

Section 2 is divided in to two main sections. The first section is the Getting Started section. This section will walk the user through using the instrument from opening the shipping case through processing downloaded data. Reading the Getting Started section will give most users enough information to starting using the instrument. Following the Getting Started section are detailed instructions for performing various procedures

General Precautions

LISST-100 is a sensitive optical instrument. Please handle it gently. Critical alignments may be disturbed if the instrument is subjected to shock or rough handling. Evidence of shock/rough handling will void the warranty. Whenever in transit, store the instrument in the provided padded shipping case.



WARNING

The LISST-100 uses a laser diode emitting a maximum of 1 mW of visible (red) light at a wavelength of 670nm. the laser beam under normal circumstances is not a threat. However, if objects are placed in the path of the laser beam, the light could be reflected into the eye causing permanent damage.

APPENDIX B

STANDARD OPERATING PROCEDURE (SOP)

HIGH VOLUME WATER SAMPLING MEDIA PREPARATION: GLASS FIBER FILTERS AND XAD-2 RESIN

FOR USE ON THE PASSAIC RIVER PILOT DREDGING PROGRAM SAMPLES

Adapted from
STL-Knoxville SOP KNOX-ID-0017
Revision 1, 11/25/02

NOTE FROM STL:
The STL SOP was developed in-house by STL and contains proprietary information and copyright is claimed.

1. Scope and Application

1.1 This procedure is applicable to the pre-sampling preparation and post-sampling extraction of glass fiber filters and XAD-2 resin sampling media used in high volume water samplers.
1.2 This procedure is designed to be used as a supplement to existing standard operating procedures that describe the preparation and analysis of extracts from environmental matrices for organic constituents. This procedure must be used in conjunction with laboratory extraction and analysis SOPs for the analyte being tested as it provides additional or alternative instructions for use when this type of sampling media is extracted and analyzed.
1.3 This procedure is applicable to the analysis of the sampling media for chlorinated dibenzo-p-dioxins (CDDs) and chlorinated dibenzofurans (CDFs), polychlorinated biphenyl (CB) congeners (PCB), and/or chlorinated pesticides, by conventional or isotope dilution gas chromatography/mass spectroscopy (GC/MS).
1.4 This procedure is restricted to use by or under the supervision of analysts experienced in the use of trace level sampling methods and the analysis of CDDs, CDFs, PCBs, and pesticides. Each analyst must demonstrate the ability to generate acceptable results with this procedure.

2 Summary of Method

2.1 This procedure describes the preparation of glass fiber filters and XAD-2 resin tubes prior to being shipped to the sampling site for high volume water sampling.
2.2 Fluoropolymer sampling tubes are washed, solvent rinsed, and baked; then packed with XAD-2 resin.
2.3 High volume water samplers operate by pumping water through glass fiber filters and tubes packed with a bed of XAD-2 resin. Filterable material in the water sample is collected on the glass fiber filters while dissolved organic contaminants are adsorbed onto the XAD-2 resin.
2.4 After sampling, the sampling media is returned to the laboratory for extraction and analysis. The glass fiber filters and the XAD-2 resin are extracted in a Soxhlet extractor using appropriate solvents for the analytes to be determined. The extracts are subjected to cleanups, if necessary to remove interferences, and are analyzed by HRGC/HRMS or GC/MS.
2.5 QC analysis checks are performed on the sampling media to verify that it is free of contamination, either by the analytes of interest or interferences, and meets the project specific requirements for cleanliness.

3 Definitions

3.1 Sampling Surrogate Standards (SS): NOT APPLICABLE
3.2 XAD-2: Sorbent resin - Amberlite XAD-2 resin. Supelco Supelpak-2, or equivalent.
3.3 Glass fiber flat filter – Glass fiber filters, without organic binder, retains particles down to 0.7 µm, Whatman GF/F 142 mm or equivalent.
3.4 Glass fiber filter cartridge – 100 mm in length, glass fibers are wound around a metal spindle 25mm in diameter, total diameter is 65 mm. Filters are baked at 385º C for 10 hours in a cross flow electric oven and wrapped in aluminum foil after cooling. Parker Filtration, Fulflo Honeycomb Filter Cartridge, Part #K39R4S-QNT.

4 Interferences

4.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferences under the conditions of analysis by performing laboratory method blanks. Analysts should avoid using PVC gloves. Gloves that are clean room approved and are a nitrile/latex blend are preferred. When rinsing glassware with organic solvents where splashing may occur a glove that resists solvents is recommended. The use of high purity reagents and solvents helps minimize interference problems.

4.2 NOT APPLICABLE

5 Safety

5.1 Procedures shall be carried out in a manner that protects the health and safety of all associates.

5.2 Eye protection that satisfies ANSI Z87.1, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately. VITON gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled.

> *Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.*

5.3 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.3.1 NOT APPLICABLE

5.3.2 Chemicals known to be flammable include methanol, acetone, toluene, hexane, nonane, and tetradecane.

5.3.3 Chemical known to be corrosive include sulfuric acid, hydrochloric acid, and potassium hydroxide.

5.4 Exposure to chemicals will be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 The preparation of all standards and reagents and glassware cleaning procedures that involve solvents such as acetone, toluene, methylene chloride, and hexane will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.6 Training: Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

5.7 Personal Hygiene: Thorough washing of hands and forearms is recommended after each manipulation and before breaks (coffee, lunch, and shifts).

5.8 Confinement: Work areas should be isolated and posted with signs. Glassware and tools should be segregated. Bench tops should be covered with plastic backed absorbent paper.

5.9 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.

5.10 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

5.11 All work must be stopped in the event of a known, or potential compromise to the health or safety of an employee. The situation must be reported immediately to a laboratory supervisor.

6 Equipment and Supplies

6.1 Glass fiber filter cartridges - Wound glass fiber on perforated core. Parker Filtration, Lebanon, Indiana (Cat # K39R4S), or equivalent.
6.2 Glass fiber filter – Whatman 142 mm GF/F glass fiber filters or equivalent.
6.3 XAD-2 Resin Tube - The teflon sampling tube is configured from components obtained from Savillex Corporation, 6133 Baker Rd, Minnetonka, MN 55345, 612/935-4100.
6.3.1 47 mm by 120 mm column segment with 1 1/2" NPT threads, P/N 504.
6.3.2 One-port transfer cap for 3/8" tubing (two each needed per tube), P/N 501-6.
6.3.3 Teflon screen with 1 mm openings (two each needed per tube), P/N 504S.
6.3.4 Teflon cloth with mesh opening .004", P/N 1105, cut into circles of 47 mm diameter (two each needed per tube).
6.3.5 Open-end cap to fit on the end of port transfer cap, SX-6.
6.3.6 Closed end cap to fit on the end of port transfer cap, SX-6C.
6.4 Wash bottles - fluoropolymer
6.5 Aluminum foil - heavy duty
6.6 Ziplock bags.
6.7 1000 mL, 2000 mL, and 4000 mL (1-L, 2-L, and 4-L) glass beakers
6.8 Syringes
6.9 Glass Soxhlet
Note: All glassware used in extraction and cleanup procedures is solvent rinsed two times before use with acetone, toluene, methylene chloride and hexane in that order. Pre-extract the soxhlet apparatus with toluene for at least four hours. Re-rinse glassware with all solvents once.
6.10 Heating mantles with temperature controls
6.11 Three-ball macro Snyder column
6.12 500 mL evaporative flask, round bottom
6.13 Glass condenser, capable of sitting on top of the soxhlet extractor
6.14 Glass wool, pre-clean with methylene chloride for a minimum of four hours in a Soxhlet. After air drying in a hood store in a glass jar with a fluoropolymer lined lid.
6.15 Nine inch borosilicate glass pipettes
6.16 Boiling beads, 6 mm glass
6.17 Rubber bulbs
6.18 40 mL vial, with fluoropolymer lined cap
6.19 Fluoropolymer boiling chips
6.20 Nitrogen blowdown apparatus (N-EVAP or equivalent)
6.21 Analytical balance, capable of weighing to 0.01 g
6.22 Tweezers and stainless steel spatulas
6.23 Micropipetters
6.24 1 mL Class A pipette
6.25 Separatory funnels, 2000 mL (2 L)
6.26 Concentrator flasks
6.27 100 mm powder funnels
6.28 Separatory funnel rotator
6.29 1-L amber jars

7 REAGENTS

7.1 Isotopically labeled sampling surrogates – NOT APPLICABLE.

7.2 XAD-2 resin - Amberlite XAD-2 resin. XAD-2 may be purchased pre-cleaned from several different vendors. If the pre-cleaned resin is not available, the laboratory must perform a cleaning procedure on XAD-2 resin. The procedure to be used is in Section 11.1.1.
7.3 Acetone – pesticide quality, or equivalent
7.4 Toluene – pesticide quality, or equivalent
7.5 Methanol – pesticide quality, or equivalent
7.6 Methylene Chloride – pesticide quality, or equivalent
7.7 Hexane – pesticide quality, or equivalent
7.8 Nonane – pesticide quality, or equivalent
7.9 Organic-free reagent water – Water run through a polishing system capable of achieving 18 Mohm resistance.
7.10 Sodium sulfate – AR Select grade or equivalent. Prepare by baking in an oven at 450º C for a minimum of four hours. After cooling, store in glass jars with fluoropolymer lined caps in the desiccator.
7.11 NOT APPLICABLE

8 Sample Collection, Preservation and Storage
8.1 Sample collection involves the operation of high volume water samplers and is beyond the scope of this SOP. Samples should be collected and shipped to the laboratory in accordance with any applicable sample collection standard operating procedures, Quality Assurance Project Plans (QAPPs), or program guidelines.
8.2 NOT APPLICABLE.

9 Quality Control
9.1 Media Cleanliness Check Sample
9.1.1 Each lot of XAD-2 resin should be checked prior to sampling media use. If the client requires resin check results, extract and analyze an aliquot of the cleaned resin along with the first batch of associated client samples. The resin must not contain any analytes or interferences at the project specific action levels. If the project specific quality objectives permit it, vendor supplied cleanliness check results may be used for pre-cleaned resins.
9.2 The collection of field blanks and other associated QC samples are the responsibility and at the discretion of the sampling collection team.
9.3 Laboratory-generated QC: NOT APPLICABLE.
9.4 Refer to laboratory quality management plan and/or associated SOPs for details of the acceptance criteria and corrective action.

10 Calibration and Standardization
10.1 None.

11 Procedure
11.1 Pre-Sampling Media Preparation
11.1.1 XAD-2 resin cleaning procedure
Note: Normally, pre-cleaned XAD-2 resin is purchased and used with no further cleanup performed. The following cleanup procedures is provide for situations in which pre-cleaned XAD-2 resin does not meet the needs of the project and further cleanup is required.

11.1.1.1 Place the XAD-2 resin in a clean beaker and rinse with reagent water. Discard the rinse. Fill the beaker a second time with reagent water and allow the resin to stand overnight. Discard this second rinse.

11.1.1.2 Place the XAD-2 resin in an all-glass thimble of a large Soxhlet extractor. The XAD-2 resin will float when in contact with methylene chloride. Therefore, add a glass wool plug on top of the resin in the thimble.

11.1.1.3 Place the thimble filled with resin into the Soxhlet extractor, add organic-free reagent water to the distilling flask, apply heat, and extract the resin for 8 hours.

11.1.1.4 Allow the Soxhlet extractor to cool, discard the water, and add methanol to the extractor. Apply heat and extract for 22 hours.

11.1.1.5 Again allowing the extractor to cool, drain off the methanol, replace it with methylene chloride. Make sure that the glass wool plug is still in place and extract for 22 hours.

11.1.1.6 Extract the resin a fourth time, using toluene as the extraction solvent, for 22 hours.

11.1.1.7 Following the toluene extraction, the XAD resin must be dried using a vacuum oven.

11.1.1.8 A quality control check should be conducted on the cleaned resin. The resin should be checked using the laboratory extraction and analysis SOPs for the analyte being tested. If the resin meets the cleanliness requirements outlined in the client’s QAPP, then the resin is acceptable to use. If analytes are found at concentrations greater that what is acceptable, the resin may be sent back for additional cleaning. An extra extraction with methylene chloride is sometimes helpful in removing the contaminants.

11.1.2 XAD-2 Resin Tube Preparation

11.1.2.1 Wash the sampling tube components (see section 6.3) with soap and water, then rinse with organic free reagent water. Bake the tube components in an oven at 150° C for one hour. Remove the tube components from the oven and allow them to cool. Rinse the tube components in order with acetone, toluene, methylene chloride and hexane and allow them to air dry. It is acceptable to place some of the smaller parts such as the closed and open-ended caps and the Teflon support screens into a beaker filled with solvent and sonicating the parts. When using the beaker, use all solvents listed above in the same order and sonicate for at least five minutes with each solvent.

11.1.2.2 Assemble one end of the sampling tube, in preparation for filling the tube, by placing the placing the support screen just inside one end of the tube and then making sure the teflon cloth is laying flat against the support screen; no XAD-2 will be able to escape the tube. Attach the one-port transfer cap and screw it onto the end of the column segment that does have "ridges". Be sure that the cloth and screen are recessed into the tube by approximately 3mm. Clamp the assembly to a ring stand or other support with the transfer cap facing down and the open end of the column that does not have the "ridges" facing up. Connect the transfer cap port to a vacuum pump with an in-line 1-L filter flask for water collection using fluoropolymer and/or latex rubber tubing. Avoid the use of laboratory tubing containing phthalate plasticizers.

11.1.2.3 Prepare a slurry by adding between 60g and 70g of resin and a large amount of organic free reagent water to a beaker. XAD-2 and water do not mix well, the slurry must be stirred frequently to keep the water and the XAD-2 mixed and keep the mixture in a slurry form. Add the resin slurry to the tube while applying vacuum to remove the water and create a flat packed bed of resin. Fill the tube with resin to within 5-7 mm of the top. Use a fluoropolymer squirt bottle filled with organic free reagent water to flatten the top of the resin bed and eliminate any gaps or channels that formed during the packing process.

11.1.2.4 Inspect the resin bed through the translucent sides of the tube to be sure that no gaps or channels are present. Remove the vacuum source, remove any XAD-2 resin from the tube and cap threads, and cap the open end of the tube with the second set of Teflon cloth, support screen, and transfer cap. Inspect the support screen to verify that an approximately 3 mm recess exists between the end of the column and the screen. Unclamp the tube assembly, place the assembly in a vise and, using the wrenches provided, tighten the caps. Finish sealing the XAD-2 tube by placing the closed end caps on both ends of the tube.

11.1.2.5 Remove the closed end cap from the end of the tube without the "ridges", add the sampling surrogate spiking solution with a syringe by gently pushing the syringe through the support screen and the teflon cloth and adding the entire contents of the syringe to the XAD-2. Replace the closed end cap.

11.1.2.6 Record the tube preparation information in a laboratory notebook.

Include the following information:

- Tube Number
- XAD Vendor
- Lot Number
- Packing Date
- Spiking Solution Identification Number
- Spiking Volume
- Spiking Date

11.1.2.7 Label the tubes with the tube number and flow direction. Wrap each tube in aluminum foil to protect from light, place them in a ziplock bag and then pack them in a cooler for shipment to the sampling site. When preparing the cooler for shipment, enclose two open-ended caps for each XAD-2 module sent with the shipment so that they can be used by the sampling team during the collection of samples.

11.1.3 Glass Fiber Filter Preparation.

Note: If QC tests on the glass fiber filters indicate that no contamination is present, the filters may be used as received. The following cleanup procedure is provided for situations in which filters do not meet the needs of the project and further cleanup is required. Document all cleanup procedures used in a sample prep log book.

11.1.3.1 Measure the filters to be certain that they meet the size requirements of the filter holders. The client should provide size requirements and measuring jigs if necessary. Set aside any filters that do not meet the size requirements of the specific project.

11.1.3.2 For the cartridge filters, place two 4000-mL beakers in a hood and fill both of them half full of methylene chloride. Unwrap the cartridge filters and place them in the first beaker (8 of them will fit, 4 on the bottom and 4 on the top). Make sure the filters are completely submerged in the solvent.

11.1.3.3 After one minute, using solvent-rinsed tweezers, pull the filters out of the first beaker and place them in the second beaker. Again, make sure the filters are completely covered by solvent.

11.1.3.4 After another one minute, soak in the second beaker, take the filters out of the beaker, and place them on aluminum foil in the hood to dry.

11.1.3.5 After the filters dry, wrap them in foil and put them in a ziplock bag. Record the date of cleaning and the lot number of the filters on the outside of the ziplock bag.

11.1.3.6 For flat glass fiber filters, place no more than 24 filters in a large glass baking dish and cover them with methylene chloride.

11.1.3.7 Allow the filters to soak for a minimum of five minutes, stirring occasionally.
11.1.3.8 Drain the methylene chloride, turn the filters over, and cover the filters with fresh methylene chloride.
11.1.3.9 After they have soaked for another five minutes, drain the methylene chloride off and spread the filters out on aluminum foil to dry.
11.1.3.10 After the filters have dried, place all the filters in a clean, solvent rinsed Petri dish and wrap with fluoropolymer tape. Place the Petri dish in a ziplock bag.
11.1.3.11 If requested by the client, a filter may be labeled and set aside to be run as a media check when the samples come back from the field for analysis.

11.2 Post-Sampling Media Extraction – NOT APPLICABLE

12 Data Analysis and Calculations
12.1 None

13 Method Performance
13.1 Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an employee who has been properly trained in its use and has the required experience.

14 Pollution Prevention
14.1 All procedures shall be conducted in a manner to minimize, as far as practical, the use of solvents, reagents and other chemicals.

15 Waste Management
15.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures.

16 References
16.1 Laboratory Quality Management Plan.
16.2 USEPA SW-846 "Test Methods for Evaluating Solid Waste" Third Edition. Methods 0010, 0023A, 3540C and 3542.
16.3 Crecelius, Eric and Lisa Lefkovitz, "HOC Sampling Media Preparation and Handling; XAD-2 Resin and GF/F Filters", Lake Michigan Mass Balance (LMMB) Methods Compendium, 1994.

GENERIC STANDARD OPERATING PROCEDURE (SOP)

HIGH VOLUME WATER SAMPLING MEDIA EXTRACTION: GLASS FIBER FILTERS AND XAD-2 RESIN

FOR USE ON THE PILOT DREDGING PROGRAM SAMPLES

SOP DATE: November 21, 2005
REVISION NUMBER: 0.3

1. Scope and Application

1.1 This procedure is applicable to the post-sampling extraction of glass fiber filters and XAD-2 resin sampling media used in high volume water samplers.
1.2 This procedure is designed to be used as a supplement to existing standard operating procedures that describe the preparation and analysis of extracts from environmental matrices for organic constituents. This procedure must be used in conjunction with laboratory extraction and analysis SOPs for the analyte being tested as it provides additional or alternative instructions for use when this type of sampling media is extracted and analyzed.
1.3 This procedure is applicable to the analysis of the sampling media for chlorinated dibenzo-p-dioxins (CDDs) and chlorinated dibenzofurans (CDFs), polychlorinated biphenyl (PCB) congeners, and chlorinated pesticides, by conventional or isotope dilution gas chromatography/ mass spectroscopy (GC/MS).
1.4 This procedure is restricted to use by or under the supervision of analysts experienced in the use of trace level sampling methods and the analysis of CDDs/CDFs, PCBs, and pesticides. Each analyst must demonstrate the ability to generate acceptable results with this procedure.

2 Summary of Method

2.1 High volume water samplers operate by pumping water through a pair of glass fiber filters (one flat filter and one cartridge filter), and tubes (cartridges) packed with a bed of XAD-2 resin. Filterable material in the water sample is collected on the glass fiber filters while dissolved organic contaminants are adsorbed onto the XAD-2 resin.
2.2 After sampling, the sampling media are shipped to the laboratory for extraction and analysis. The glass fiber filters and the XAD-2 resin are separately extracted in a Soxhlet extractor using appropriate solvents for the analytes to be determined. The extracts are subjected to cleanups, if necessary to remove interferences, and are analyzed by HRGC/HRMS or GC/MS.
2.3 QC analysis checks are performed on the sampling media to verify that it is free of contamination, either by the analytes of interest or interferences, and meets the project specific requirements for cleanliness.

3 Definitions

3.1 Sampling Surrogate Standards (SS): Surrogates which are added to the sample media prior to the shipment of the sampling media to the field. Sampling surrogates are not used for this task order.
3.2 XAD-2: Sorbent resin - Amberlite XAD-2 resin. Supelco Supelpak-2, or equivalent.
3.3 Glass fiber flat filter – Glass fiber filters, without organic binder, retains particles down to 0.7 µm, Whatman GF/F 142 mm or equivalent.
3.4 Glass fiber filter cartridge – 100 mm in length, glass fibers are wound around a metal spindle 25mm in diameter, total diameter is 65 mm. Filters are baked at 385º C for 10 hours in a cross flow electric oven and wrapped in aluminum foil after cooling. Parker Filtration, Fulflo Honeycomb Filter Cartridge, Part #K39R4S-QNT.
3.5 Sample. Each sample will consist of either (a) a set of glass fiber filters, consisting of one GFF flat filter and one GFF cartridge, or (b) a pair of XAD-2 resin cartridges. Do not combine GFF and XAD sample media.

4 Interferences

4.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferences under the conditions of analysis by performing laboratory method blanks. Analysts should avoid using PVC gloves. Gloves that are clean room approved and are a nitrile/latex blend are preferred. When rinsing glassware with organic solvents where splashing may occur a glove that resists solvents is recommended. The use of high purity reagents and solvents helps minimize interference problems.

4.2 Interferences co-extracted from the samples will vary considerably from matrix to matrix. CDDs and CDFs are often associated with other interfering chlorinated substances such as polychlorinated diphenyl ethers, polychlorinated naphthalenes, and polychlorinated alkyldibenzofurans.

5 Safety

5.1 Procedures shall be carried out in a manner that protects the health and safety of employees.

5.2 Eye protection that satisfies ANSI Z87.1, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately. Viton gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled.

> *Note: Viton is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.*

5.3 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.3.1 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: benzene and methylene chloride, 2,3,7,8-TCDD and all other 2,3,7,8- substituted CDDs or CDFs.

> *Note: The 2,3,7,8-TCDD isomer has been found to be acnegenic, carcinogenic, and teratogenic in laboratory animal studies. Other CDD/CDFs containing chlorine atoms in positions 2,3,7,8 are known to have toxicities comparable to that of 2,3,7,8-TCDD. The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.*

5.3.2 Chemicals known to be flammable include methanol, acetone, toluene, hexane, nonane, and tetradecane.

5.3.3 Chemicals known to be corrosive include sulfuric acid, hydrochloric acid, and potassium hydroxide.

5.4 Exposure to chemicals will be as low as reasonably achievable. It is recommended that samples be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Keep solvent and waste containers closed unless transfers are being made.

5.5 The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as acetone, toluene, methylene chloride, and hexane should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.6 Training: Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.
5.7 Personal Hygiene: Thorough washing of hands and forearms is recommended after each manipulation and before breaks (coffee, lunch, and shifts).
5.8 Confinement: Work areas should be isolated and posted with signs. Glassware and tools should be segregated. Bench tops should be covered with plastic backed absorbent paper.
5.9 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.
5.10 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.
5.11 Work must be stopped in the event of a known, or potential compromise to the health or safety of an employee. The situation must be reported immediately to a laboratory supervisor.

6 Equipment and Supplies

Note: Not all the items listed below will be required under this Task Order. Items 6.1, 6.2, and 6.3 are the sample apparatus/media; these will be prepared by others and will be submitted as the samples for this Task Order.

6.1 Glass fiber filter cartridges - Wound glass fiber on perforated core. Parker Filtration, Lebanon, Indiana (Cat # K39R4S), or equivalent.
6.2 Glass fiber filter – Whatman 142 mm GF/F glass fiber filters or equivalent.
6.3 XAD-2 Resin Tube - The teflon sampling tube is configured from components obtained from Savillex Corporation, 6133 Baker Rd, Minnetonka, MN 55345, 612/935-4100.
6.3.1 47 mm by 120 mm column segment with 1 1/2" NPT threads, P/N 504.
6.3.2 One-port transfer cap for 3/8" tubing (two each needed per tube), P/N 501-6.
6.3.3 Teflon screen with 1 mm openings (two each needed per tube), P/N 504S.
6.3.4 Teflon cloth with mesh opening .004", P/N 1105, cut into circles of 47 mm diameter (two each needed per tube).
6.3.5 Open-end cap to fit on the end of port transfer cap, SX-6.
6.3.6 Closed end cap to fit on the end of port transfer cap, SX-6C.
6.4 Wash bottles - fluoropolymer
6.5 Aluminum foil - heavy duty
6.6 Ziplock bags.
6.7 1000 mL, 2000 mL, and 4000 mL (1-L, 2-L, and 4-L) glass beakers
6.8 Syringes
6.9 Glass Soxhlet
Note: All glassware used in extraction and cleanup procedures is solvent rinsed two times before use with acetone, toluene, methylene chloride and hexane in that order. Pre-extract the soxhlet apparatus with toluene for at least four hours. Re-rinse glassware with all solvents once.
6.10 Heating mantles with temperature controls
6.11 Three-ball macro Snyder column
6.12 500 mL evaporative flask, round bottom
6.13 Glass condenser, capable of sitting on top of the soxhlet extractor
6.14 Glass wool, pre-clean with methylene chloride for a minimum of four hours in a Soxhlet. After air drying in a hood store in a glass jar with a fluoropolymer lined lid.
6.15 Nine inch borosilicate glass pipettes

6.16 Boiling beads, 6 mm glass
6.17 Rubber bulbs
6.18 40 mL vial, with fluoropolymer lined cap
6.19 Fluoropolymer boiling chips
6.20 Nitrogen blowdown apparatus (N-EVAP or equivalent)
6.21 Analytical balance, capable of weighing to 0.01 g
6.22 Tweezers and stainless steel spatulas
6.23 Micropipetters
6.24 1 mL Class A pipette
6.25 Separatory funnels, 2000 mL (2 L)
6.26 Concentrator flasks
6.27 100 mm powder funnels
6.28 Separatory funnel rotator
6.29 1-L amber jars

7 REAGENTS

7.1 Isotopically labeled sampling surrogates – Not applicable to this Task Order. (Note that all spiking compounds required by the analytical methods for CDD/CDFs, CB congeners, and pesticides, must be added.)
7.2 XAD-2 resin - Amberlite XAD-2 resin. The work covered by this task order does not include provision of XAD-2 resin or resin cartridges; these will be provided by the sampling organization.
7.3 Acetone – pesticide quality, or equivalent
7.4 Toluene – pesticide quality, or equivalent
7.5 Methanol – pesticide quality, or equivalent
7.6 Methylene Chloride – pesticide quality, or equivalent
7.7 Hexane – pesticide quality, or equivalent
7.8 Nonane – pesticide quality, or equivalent
7.9 Organic-free reagent water – Water run through a polishing system capable of achieving 18 Mohm resistance.
7.10 Sodium sulfate – AR Select grade or equivalent. Prepare by baking in an oven at 450º C for a minimum of four hours. After cooling, store in glass jars with fluoropolymer lined caps in the desiccator.
7.11 Standard solutions are purchased in stock solutions from Cambridge Isotope Laboratories and Wellington Laboratories. Working standard solutions are prepared by diluting the stock solutions into nonane. For spiking solution analytes, concentrations and volumes see the appropriate extraction and analysis SOPs.

8 Sample Collection, Preservation and Storage

8.1 Sample collection involves the operation of high volume water samplers and is beyond the scope of this SOP. Samples will be collected and shipped to the laboratory in accordance with applicable sample collection standard operating procedures and the project-specific QAPP.
8.2 For sample storage and holding time requirements, see the appropriate laboratory extraction and analysis SOPs for the analyses being performed or the project-specific QAPP.

9 Quality Control

9.1 Media Cleanliness Check Sample

9.1.1 A blank XAD-2 resin will be submitted by the sampling organization and designated for blank analysis. Extract and analyze an aliquot of the cleaned resin (i.e., cartridge blank) along with the first batch of samples. The resin must not contain any analytes or interferences at the project specific action levels.

9.2 The collection of additional field blanks and other associated QC samples are the responsibility and at the discretion of the sampling collection team.

9.3 Laboratory-generated QC:

9.3.1 Method blanks – A laboratory method blank must be prepared with each analytical batch of 20 or fewer environmental samples.

9.3.1.1 For GFF samples, the method blank consists of clean sand.

9.3.1.2 For XAD resin samples, the method blank consists of clean sand.

9.3.2 Laboratory Control Sample/Duplicate (LCS/LCSD) – An LCS and LCSD (if needed) is prepared with each analytical batch of 20 or fewer samples. The LCS/LCSD are spiked with the PAR compounds listed in the SOPs for analysis for those specific compounds. The matrix (material) used for the LCS/LCSD must be the same as used for the method blank.

9.3.2.1 For GFF samples, the LCS/LCSD consists of a clean glass fiber filter; this will be provided by the field sampling team and identified as clean matrix for LCS on the COC.

9.3.2.2 For XAD resin samples, the LCS/LCSD will consist of clean resin cartridge(s); this will be provided by the field sampling team and identified as clean matrix for LCS on the COC.

9.4 Refer to laboratory quality management plan and/or associated SOPs for details of the acceptance criteria and corrective action.

10 Calibration and Standardization

10.1 None.

11 Procedure

11.1 Pre-Sampling Media Preparation

NOTE: Section 11.1 is not applicable to non-RAS CLP Laboratories analyzing samples submitted under this task order and has been deleted.

11.2 Post-Sampling Media Extraction

11.2.1 XAD-2 Resin sample extraction

11.2.1.1 Inspect the XAD tube. Make note of the appearance of the tubes including any differences in color or texture of the resin.

11.2.1.2 Assemble a clean and rinsed extra large Soxhlet extractor and 1-L round bottom flask. Place a glass wool plug in the bottom of the extractor body to cover the inlet of the siphon tube to prevent sample material from being siphoned into the round bottom while the samples are being extracted.

11.2.1.3 Carefully remove the closed end cap, the one port transfer cap, the support screen, and the teflon cloth from the side without the "ridges". The XAD-2 is usually very wet so caution needs to be used when disassembling the resin tube so that no water and XAD-2 are lost.

11.2.1.4 Remove about 15 grams of resin from the tube and transfer it to the soxhlet extractor. Add acetone to the tube and slurry the remaining resin out of the tube and into the Soxhlet extractor.

11.2.1.5 Take the closed end cap off the other end of the resin tube. Rinse any remaining resin out of the tube with acetone and into the Soxhlet to complete the transfer of resin to the extractor.
11.2.1.6 Add enough additional acetone to the body of the extractor to causes the Soxhlet to cycle, catching the acetone/water mixture in the round bottom flask.
11.2.1.7 Rinse any resin that is clinging to the sides of the extractor down into the main body of the extractor body with acetone. Place a glass wool plug on top of the resin.
11.2.1.8 Remove the round bottom flask and transfer the acetone/water mixture to a 1-L amber bottle. Put aside for later use.
11.2.1.9 Assemble two additional Soxhlet extractors for each batch and place a glass wool plug and a glass fiber filter in the extractors for the method blank and LCS (OPR).
11.2.1.10 Three analytical methods are requested on each sample for the Passaic Pilot Dredge samples (CDD/CDFs, PCB congeners, and pesticides). Consult the special analysis instructions and project specific instructions to determine the analytical method to be performed on the XAD. Add 1.0 mL of the internal standard spiking solutions as required by each requested method. If the extracts are to be split for multiple analyses, the spike amount is divided by the split ratio.
Example: If the extract is to be split for CDD/CDF, PCB congener, and pesticide analysis, the split ratio for each analysis is 0.333. Dividing 1.0 mL of internal standard spiking solution by the split ratio (0.333), results in a spike volume of 3.0 mL.
11.2.1.11 Spike the sample in the Soxhlet designated as the LCS (OPR) with 1.0 mL of the PAR (native) spiking solution or an appropriate amount if split extracts are required by each analytical method.
11.2.1.12 Add 700 mL of acetone:hexane (1:1) to the empty round bottom flask and reassemble the Soxhlet extractor. Place it in the heating mantle and attach a condenser. Extract the XAD for 16 hours after adjusting the heating mantle to achieve a Soxhlet cycling rate of at least five times per hour.
11.2.1.13 Turn off the mantles and allow the extracts to cool. Remove the Soxhlet and add a couple of fresh boiling chips. Place a Snyder column on top of the round bottom and reduce the volume to approximately 300 mL. Transfer the remaining solvent in the round bottom flask to the 1-L amber bottle containing the acetone/water mix. Rinse the round bottom flask twice with acetone and twice with hexane.
11.2.1.14 Go to section 11.2.1.16.

> *Note: Not only does the acetone/water contain a significant amount of water, so does the acetone/hexane extract. To remove the water completely, do a separatory funnel extraction, adding enough water to remove all the water and acetone from the extract.*

11.2.1.15 For CDD/CDF analysis on the XAD, reattach the round bottom flask filled with 700 mL of toluene to the Soxhlet extractor and continue with a second 16-hour extraction, adjusting the heating and cycle rate as before. After the extraction is complete, concentrate the toluene extract to approximately 10 mL and transfer to a vial marked at 10 mL, rinsing the round bottom flask three times with 3 mL of toluene and adding these rinsings to the 40 mL vial, then setting this vial aside for later use.
11.2.1.16 Set up enough separatory funnels for every extract.
11.2.1.17 Transfer the sample extracts from the 1-L bottles into the separatory funnel. Rinse the bottle once with acetone and once with methylene chloride and add these rinsings to the separatory funnel.
11.2.1.18 At this point, enough water needs to be added to separate the acetone and the hexane. Usually 750 mL is enough, but if there is not a clear separation, add 200 mL increments of

additional deionized water until there is a clear separation. The hexane should appear as a layer on top of the acetone/water mix; there will be a small layer of methylene chloride at the bottom because of the rinse of the extract bottle added with methylene chloride.

11.2.1.19 Rinse a concentrator flask and a 100 mm glass funnel. Plug the funnel with pre-cleaned glass wool and fill the funnel 2/3 full with sodium sulfate. Rinse the funnel and the sodium sulfate with methylene chloride. After the methylene chloride stops dripping from the funnel, place the funnel on top of the concentrator flask. A paper clip may be needed to aid in the filtering.

11.2.1.20 Filter the methylene chloride through the sodium sulfate and into the concentrator flask. Then drain the water/acetone mix back into the 1-L amber bottle. (An additional container may be necessary due to the additional volume added to the separatory funnel.) Then filter the hexane through the sodium sulfate and into the concentrator flask. Rinse the sodium sulfate once with hexane.

11.2.1.21 Add all the water and acetone back to the separatory funnel. Add 60 mL of methylene chloride to the bottle that contained the sample extract. After swirling the methylene chloride in the bottle, carefully add the methylene chloride to the separatory funnel.

11.2.1.22 Extract by shaking the separatory funnel for two minutes or by placing and securing the separatory funnel to a separatory funnel rotator and rotating for two minutes.

> *CAUTION: Care should be used while performing this operation. Vent the separatory funnel frequently. Goggles should be worn when performing this procedure.*

11.2.1.23 Allow the water and the methylene chloride to separate for 10 minutes. If it is not separated after 10 minutes, try to break up the emulsion by gently swirling the sample or tilting the separatory funnel on its side.

11.2.1.24 Drain the methylene chloride from the separatory funnel into the glass funnel that is filled with sodium sulfate, allowing the extract to drip into the round bottom flask or concentrator tube. Do not to allow water to escape the separatory funnel or the sodium sulfate will harden and block the flow of the extract. When an emulsion is present, do not drain the emulsion until the third methylene chloride shake has been completed.

11.2.1.25 Repeat steps 11.2.1.21 through 11.2.1.24 two more times.

11.2.1.26 After the third methylene chloride portion has filtered through the sodium sulfate, rinse the funnel with approximately 40 mL of methylene chloride.

11.2.1.27 Remove the separatory funnel from the hood and pour the extracted water/acetone mix into the extracted waters waste carboy.

11.2.1.28 The methylene chloride/hexane extract is now concentrated with the Labconco Rapidvap system or a heating mantle and Snyder column.

11.3 Rapidvap Concentration

11.3.1 Preheat the unit to the appropriate temperature for the solvent used in the extraction.

11.3.2 Set the operating parameters on the programmer. For example, if the there is 300 mL of a methylene chloride extract, the following parameters may be used:

- Temperature: 35° C
- Vortex speed: 50%, to be increased at a later time
- Nitrogen: 9 psi
- Timer set: 40 minutes

11.3.3 Place 600 mL concentrator tubes containing the extract in the Rapid-Vap. Begin concentrating the extract, adjusting the vortex speed for the proper rate of concentration.

11.3.4 When the extract has been concentrated to approximately 20 mL, add approximately 60 mL of hexane. Concentrate the extract to a final volume of approximately 10 mL. Shut off the nitrogen flow and turn off the Rapid-Vap or remove the 600 mL concentrator tube to prevent further concentration.

11.3.5 Transfer the extract with a disposable pipette to a 40 mL vial marked at 20 mL, rinsing the sample tube three times with 3 mL of hexane.

11.4 Snyder column concentration step.

11.4.1 Remove the glass funnel from the top of the round bottom flask, attach a Snyder column to the round bottom flask and place in a heating mantle. Pre-wet the Snyder column by adding approximately 1 mL of solvent through the top. Adjust the heating mantle as required to achieve the proper rate of distillation. At the proper rate, the balls of the column will actively chatter but the chambers will not flood.

11.4.2 Once the sample is concentrated down to approximately 20 mL, add approximately 60 mL of hexane and concentrate to approximately 10 mL.

11.4.3 Transfer the extract with a disposable pipette to a 40 mL vial marked at 20 mL, rinsing the round bottom flask three times with 3 mL of hexane.

11.4.4 At this point in the procedure, if the extracts are being analyzed for multiple analyses, the extracts must be split into portions for each separate analysis. Consult the special analysis instructions and project specific instructions to determine the ratio to be used for the split. Normally, the extract will be split into equal portions for each analysis; however in some cases, a higher split ratio may be used for some analyses to provide lower detection limits. No archive portion is required for this task order.

11.4.5 Adjust the volume in the 40 mL vial to 20 mL. Split the 20 mL hexane extract into the required number of portions at the proper ratio. Transfer each portion to a clean 40 mL vial and label it with the proper sample and fraction identification. Record the split ratios and volumes on the sample prep bench sheets.

11.4.6 If a toluene extraction was performed for CDD/CDF analysis, concentrate the toluene extract to 10 mL and split the 10 mL toluene extract in the same manner as the hexane extract. Combine the hexane and toluene portions that are identified for the dioxin/furan analysis only and concentrate to near dryness and exchange into hexane. Archive the remaining portion of toluene.

11.4.7 Proceed to cleanup the extracts using the procedures appropriate for each specific analytical method. Extracts for dioxin/furan analysis may be subjected to acid/base wash, silica gel/alumina dual column chromatography, carbon column chromatography, followed by a second silica gel/alumina dual column chromatography. Extracts for PCB congener analysis may be subjected to acid wash followed by florisil column chromatography, and a sulfur clean-up if needed. Extracts for pesticide analysis may be subjected to silica gel column chromatography and a sulfur clean-up if needed. Document all cleanups performed on the sample prep bench sheets.

11.5 Glass Fiber Filter sample extraction

11.5.1 Place a large glass wool plug in the bottom of the Soxhlet to prevent particulate matter from migrating into the round bottom during extraction. Place the glass fiber filter along with any additional filters into the body of a Soxhlet extractor. The flat filters should be placed in the Soxhlet first.

Note: The filters are usually very wet; fill up the Soxhlet with acetone and cause it to cycle into the round bottom and then place the acetone/water portion in a 1-L amber jar, similar to what was done to the XAD-2. (See sections 11.2.1.6 through 11.2.1.8.)

11.5.2 Extract the glass fiber filters in a Soxhlet extractor, concentrating it, and removing of the water in the sample extract by separatory funnel techniques following the same procedures used for XAD-2 resin in section 11.2.1 through 11.3.5.

11.6 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure, except those specified by project specific instructions, shall be completely documented using a Nonconformance Memorandum and approved by appropriate technical and senior/ management staff as specified in the laboratory's Quality Management Plan. The client shall be notified of any procedural variations implemented and documentation shall be provided in the Case Narrative.

11.7 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12 Data Analysis and Calculations

12.1 None applicable to this extraction SOP.

13 Method Performance

13.1 Training Qualification: It is the group/team leader's responsibility that this procedure is performed by an employee who has been properly trained in its use and has the required experience.

14 Pollution Prevention

14.1 These procedures shall be conducted in a manner to minimize, insofar as practical, the use of solvents, reagents and other chemicals.

15 Waste Management

15.1 Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedures.

16 References

16.1 Laboratory Quality Management Plan.

16.2 USEPA SW-846 "Test Methods for Evaluating Solid Waste" Third Edition. Methods 0010, 0023A, 3540C and 3542.

16.3 Crecelius, Eric and Lisa Lefkovitz, "HOC Sampling Media Preparation and Handling; XAD-2 Resin and GF/F Filters", Lake Michigan Mass Balance (LMMB) Methods Compendium, 1994.

16.4 USEPA Analytical Services Branch (ASB) Statement of Work for Analysis of Chlorinated Biphenyl (CB) Congeners, Multi-Media, Multi-Concentration. CBC01.0, May 2005

16.5 USEPA Analytical Services Branch (ASB) Statement of Work for Analysis of Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs), Multi-Media, Multi-Concentration. DLM02.0, May 2005

16.6 USEPA Contract Laboratory Program Statement of Work for Organics Analysis, Exhibit D – Analytical Method for Pesticides. Chlorinated Biphenyl (CB) Congeners, Multi-Media, Multi-Concentration. SOM01.1, May 2005.

17 Miscellaneous

17.1 This procedure is generic and must be adapted to the specific laboratory performing the work.

APPENDIX C

USGS-SOP #1.
General Procedures for Preparing for Sampling Event.

1.0. General.

The working area of each watercraft used in this work will be washed down with clean tap water and allowed to dry before the sampling. A work area will be set up near the TOPS sampler consisting of a new layer of plastic sheeting that will be taped down. Before handling filters and XAD columns, a piece of clean aluminum foil will be placed in a stainless steel flat pan, which will allow filters and columns to be placed down (if necessary) while installing, removing and wrapping. Filter holders, tongs, and other handling equipment will be kept wrapped in foil and stored in plastic bags when not in use.

2.0 TOPS Cleaning

The TOPS sampler will be cleaned and refurbished in the USGS NJ District Lab before prior to use for the Passaic Pilot Dredge Program sampling event.

The TOPS filter canister and inline filter holder will be removed and washed in hot soapy water and cleaned with a soft bristle brush and rinsed with tap water. The inline filter holder plate will be sonicated in hot soapy water for 30 minutes. The connecting inlet and outlet tubing will be disconnected and inlet and outlet tubing dedicated for cleaning purposes only will be used. A 5 L carboy of hot, soapy (1% Alconox) water will be pumped through the TOPS sampler at a rate which cycles through the unit every 10 minutes (2.5 L/min), for a total of at least 30 minutes. The internal sampling chamber of the TOPS samplers will be washed with hot soapy water and rinsed with clean tap water, taking care not to get the electronic or electrical parts wet. The soap wash will be followed by a rinse of 5 L of clean tap water that is cycled through the TOPS for 30 minutes. The rinse water will be drained, and a second 5 L rinse of tap water will be cycled through the unit for 30 minutes. At this point the pump tubing will be replaced with new tubing, and 5 L of deionized laboratory water will be pumped through the TOPS. The filter canister and inline filter holders will be rinsed with GC grade methanol and then rinsed with DI water. The canister will be replaced in the TOPS and tightened down, and the inline filter holders will then be wrapped in foil and double-bagged for transport to the field.

3.0 ISCO Cleaning

The sample bottle cage will be removed from the ISCOs and wiped clean using clean tap water. The ISCOs storage chamber will be washed in hot soapy water, rinsed with clean tap water and dried. The sample holding apparatus will then be reinstalled. A new piece of pump tubing, distributor tubing, and connection tubing will be installed before each day. All tubing will be acid-washed (see procedure in Table 1.2 below) and rinsed with de-ionized water in the laboratory and bagged for transport to the field.

Once set up in the field, the ISCO samplers will be checked for proper operation. This check will be done by pumping 500 mL of river water into each of four bottles, having the sampler automatically switch bottles between samples. A program for executing this test is listed in Table 1.3. The volume of water delivered to each bottle will then be measured using a graduated cylinder, and if necessary, the ISCO pump will be recalibrated using **“CALIBRATE PUMP”** under the selection **“MANUAL FUNCTIONS”**. If an ISCO pump does not appear to be functioning properly, it will be recalibrated or removed and returned to the District laboratory for repair, and replaced with a spare ISCO sampler.

Table 1.2 Washing Procedure for ISCO tubing and fittings

Tubing:

1. Set up peristaltic pump. Use a piece of MasterFlex tubing with fitting on one end.
2. Run de-ionized water through sample tubing for 20 minutes as initial rinse.
3. Rinse off outside of tubing with de-ionized water (about 1-2 min).
4. Fill a bath with phosphate-free laboratory detergent and water to make a soapy solution.
5. Attach end of sample tubing to MasterFlex tubing.
6. Put the tubing ends into the soapy bath.
7. Run the soapy water through the tubing for at least 20 minutes.
8. Dump out soapy water when finished.
9. Hook up de-ionized water hose to the end of the sample tubing, run for 20 minutes to flush out any soap.
10. Fill a designated acid tub with 3 inches of de-ionized water and pour one bottle (800 mL) of HCl into the tub. Continue to fill with de-ionized water until you have a 10% acid solution.
11. Hook up the tubing the same way as for the soapy bath, and let run for 20 minutes.
12. When done, neutralize the acid solution in the tub with baking soda and pour down drain. Leave faucet on to flush.
13. Attach the de-ionized water hose to the end of the sample tubing and run for 20 minutes to flush.
14. Detach de-ionized water hose and attach MasterFlex tubing to the end of the sampling tubing. Use the pump to get out any standing water in the tubing. You can also do a nitrogen purge to get rid of excess water.
15. Double bag tubing and freeze.

Fittings:

1. Designate a plastic container with lid for cleaning; add 5% HCl solution.
2. Add fittings and make sure they are completely submerged in solution. (Note: Stainless steel fittings are not subject to acid wash; these parts are washed in hot soapy tap water, rinsed in DI water, sonicated as needed, solvent rinsed, DI rinsed, allowed to dry, and wrapped in aluminum foil for transport.)
3. Let this sit for about 20 minutes.
4. Rinse thoroughly with de-ionized water.
5. When this is complete, dry the fittings on the drying rack. Double bag fittings and freeze when dry.

Table 1.3 Calibration Program for checking ISCO samplers.

Turn on ISCO
Ensure Sampler reads Extended Programming
Enter "Program"
Change Name? No (Not necessary for simple calibration program that will be overwritten)
Change Site Description? No
Select Units for Length: ft
Number of Bottles: Enter 4
Bottle Volume is: 4.00 lit
Warning Standard Bottle Volume Exceeded, Are you sure? Enter "Yes"
Suction Line Length is: Enter proper length per station
Suction Head of: Enter proper head per station
Enter "One Part Program"
Enter "Uniform Time Paced"
Time between sample events: Enter 3 minutes
Enter "1 Bottle Per Sample Event"
Switch Bottles on: Number of Samples
Switch Bottles every: 1 Sample
Run continuously? Enter "No"
Take (Enter 4) samples
Do you want samples dependent on flow? Enter "No"
Sample Volume: Enter 500 ml
Enable: none
Once Enabled, Stay Enabled? No
Sample at Disable? No
Sample at Enable?   No
Reset Sample Interval at Enable? No
Continue Countdown while Disabled? No
Enter "Done" at Pause and Resume screen
Enter "No Delay to Start"
Programming Complete, Run This Program Now? Enter No
Select "Other Functions"
Select "Hardware"
Use Liquid Detector? Yes
Do you have a rain gage connected? No
YSI 600? No
Master/Slave? No
Bottle Full Detect? Yes
Select "Continuous Backlight"
Event Mark sent for: Complete Samples
Pre-sample Purge: 200 counts or depending on setup of station 15 if line is pressurized and continuous
Post Sample Purge: Dependent on Head
Set I/O1: none, Set I/O2: none. Set I/O3: none
Analog Output Hardware has not been installed

Table 1.3 continued. Calibration Program for checking ISCO samplers.

Periodic Serial Output? No
Dialout I/O1 Becomes True? No, I/O2? No, I/O3? No
Dialout Numbers: Enter three times
1 Minute Delay Between Calls

Now the moment you have been waiting for: Run Program!

After program is finished, remove ISCO top portion and measure volume in each bottle using a graduated cylinder. If volume is significantly off (Program Volume and Actual Volume differ by more than 1% or 5ml) then this procedure to calibrate the sampler must be followed:

Select “Other Functions”
Select “Manual Functions”
Select “Calibrate Volume”
Sample Volume: 500 ml

Press the Enter Key when you are ready (Make sure you have disconnected the tubing going into the base of the sampler and have positioned it into a graduated cylinder.)

Once the sample has been taken it asks you—Volume Delivered At which point you read the volume off the cylinder and enter the value. If this value differs significantly from what ISCO thought it pumped, it may ask you some question along the lines of Are you crazy? Just enter Yes and Repeat the process two or three more times. This should fix all problems and the above calibration program should be rerun.

If above procedure does not alleviate the problem, one last resort is to reset the machine by unplugging the battery outlet for 30 seconds, holding down the red stop key and enter for 30 seconds while re-powering the sampler. All settings should be erased and returned to factory settings. If this doesn’t work, the sampler should be returned to the lab and use a spare ISCO, or call an ISCO at 1-800-228-4373 and ask for a service technician.

4.0 Equipment Inventory

The equipment in each watercraft will be inspected and inventoried before and after each sampling day. The equipment check list will be recorded and included in the field notes returned after the sampling event. Any deficiencies or broken equipment will be fixed or replaced prior to the next sampling event. (The equipment check list will be developed after the sampling vessels have been made available and the equipment is assembled and placed in the vessels.)

USGS-SOP #2.
Logbooks and Forms

1.0 General

Each watercraft station will have a logbook (waterproof notebook) in which all field data and notes for each day will be recorded along with any information regarding the equipment and operations. The logbook will remain with the sampler operator and in the watercraft at all times, in case a sampling crew is replaced. At the completion of the event, the primary sampler will re-check that the forms are completed, return to the office the field data forms, make copies of these forms for the records, and return the original data sheets to the Project Chief who will check and archive the sheets.

Chain-of-Custody Record (COC). A chain-of-custody (COC) form suitable for the TOPS and ISCO samples is reproduced below as Form #2. This form will be used in addition to any form required and supplied by the NJDOT. Each researcher will complete and sign the form at the time samples are collected and immediately before shipping to the contract laboratory. One copy will be kept by the project chief. The contract laboratory will initialize the form and fill in the "Condition on Receipt" column, and will then send one copy back to the project chief (included with the sample data result package). In the event that an COC form is found not to be complete upon receipt of the samples, the laboratory will store the sample and attempt to contact the Project Chief for the missing information. Once the COC is complete, the samples will be inventoried and prepared for analysis.

NJDOT will provide any additional chain-of-custody forms required to accompany each sample within a cooler (XAD columns, GFFs and bottle sets, TSS and POC samples).

In addition to the COC, other forms were constructed to provide a written record of the sampling effort. These forms are reproduced below. The forms will be copied onto self-adhesive paper and placed in the sampler log book for storage.

TOPS sampler log form. For each TOPS sampler employed in the field, a daily TOPS sampling field data log will be kept by the operator (Forms 2.3 and 2.4). A new form will be completed for each TOPS sample set (i.e., two forms will be produced if two TOPS samples are collected in a single day).

ISCO 1 and ISCO 2 Sample Log. A sample log sheet will be filled out for each half-day of sampling (Form 2.4). This table is used to record the SS and POC samples that are collected. One sample log will be prepared each half day of sampling.

Sample Identification Log. At the conclusion of each day, a sample identification sheet (Form 2.5) will be completed to identify the samples collected on each day. One form will be produced each day.

The sampling program that will be used for the ISCO samples for SS (ISCO 1) and for POC (ISCO 2) are listed as Form 2.6. A copy of this program will be kept in the boat with the field note.

Form 2.1. Chain of Custody from USGS to Labs

NJDEP New Jersey Toxics Reduction Work Plan
Contaminant Assessment Reduction Program
Office of Coastal Planning & Program Coordination
PO Box 418 Trenton, NJ 08625
609-292-2662 (T) jpecchio@dep.state.nj.us

Pg 1 of 1

External Chain-of-Custody NJTRWP-4 Study I-C, I-D, I-E

Survey ID:_____________ Research Group: ___ USGS ___SIT ___SIT-Rutgers
USGS-KY Sediment Samples only: Account # for Billing: 4434-16803
Lead Sampler: _________________________ Phone:___________________

Signature:_____________________________ Fax: ______________________

Number	**Sample ID** *Number & suffix* SA= sample FB= field blank TB= trip blank EB= equip. blank	Field Station ID	Date Sampled	Time Sampled	XAD Column Number #1 spiked	XAD Column Number #2 unspiked	**Sample Matrix** X-XAD W- water FP- filter pack (TOPS) F-POC filter	Number of Containers	Sample Volume (L)	XAD NJXAD	Filter NJFilter	PAH Water NJPAHB	Metals NJMetals T/D/H	Metals NJMEtals B	DOC	POC	SS -- Analysis Code Enter "1"	Completed by Lab	Container Intact/Sufficient Volume	Label Intact
																			__Y__N	__Y__N
																			__Y__N	__Y__N
																			__Y__N	__Y__N
																			__Y__N	__Y__N
																			__Y__N	__Y__N
																			__Y__N	__Y__N
																			__Y__N	__Y__N
																			__Y__N	__Y__N
																			__Y__N	__Y__N
																			__Y__N	__Y__N

Relinquished By (Sign)	Received By (Sign)	Date	Time

Certification of COC & Preservation

Certification of Acceptable COC and preservation:
Name: __________________________
Signature: Date:

Laboratory Log-In / Preservation Check Sheet

Sample Cooler Received by Laboratory_____________________________(name/location)
Sample Custodian Receipt: _____ - _____ - _____ (date) _____:_____ (time)
Custody Seal Intact: ____ Yes _____ No
Name (print) ________________________ Signature: _________________________
Laboratory COC Initiated: _____ - _____ - _____ (date) Time: _____:_____
Ice present in sample cooler? _____Yes _____ No
Temperature of control bottle: _______ C^{o}
Note: If the temperature control bottle is >6^{o}C, contact the USGS or GLEC Project Manager before analysis.

Sample Shipment from Principal Investigator to Analytical Laboratories
Custody Seal Number:__________________________________
Temperature of Control Bottle: _____ ^{o}C
Shipping Container Sealed: _____ - _____ - _____ (date)
Shipped: _____ - _____ - _____ (date)
Carrier: ________________________________
Relinquished By:
Name: ___________________________Signature:_____________________

Form 2.2. TOPS Sampling Field Data Form

Site Name: ________________ Sampler: ________ TOPS ID Number:_________ TOPS Cleaned Date:_____________
Survey #:_______________ Date/time of enable: ___________________ Mflex Setting:________ FMI setting:________
Date/time of Shutdown:____________ Cycle # START:_____________ Cycle # FINISH:___________ TOTAL Cycles:________

Canister Filter Number________ Total volume passed________

Cycle #	Date	Time	FMI Time	FMI Grab sample volume (mL)	FMI Grab sample time	FMI Grab flow rate (ml/min)	Waste Meter total flow	Waste Meter flow rate	Waste Grab sample volume (mL)	Waste grab sample time	Waste Grab flow rate (l/min)

XAD Column Numbers for Field Blanks ________:_______:_________(s) ________:_______:_________ (us) Equipment Blank? ________:_______:_________
Comments:____

Form 2.2. TOPS Sampling Field Data Form (continued)

Station:________________________ Date:___________ Survey #:______________________ TOPS ID#__________

XAD Columns

Date Installed : __________ FMI time: _______ 1st column #: ____________ spiked: Y N 2nd column #: __________
Date Removed:___________ FMI time:________
Date Installed : __________ FMI time: ________1st column #: ___________ spiked: Y N 2nd column #: _________
Date Removed:___________ FMI time:________

GFF : Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______

Flat Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Filter: Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______
Date: ___________ FMI time start: __________ Time start:________ FMI time finish: _________ Time finish:_______

Form 2.3 TOPS SAMPLING FORM—TOTAL VOLUMES COLLECTED

FMI Volume Drained :	Date/Time Drained:
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	
9.	
10.	
Total Waste Volume:	**Date/Time Drained:**
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	

TOTAL FMI VOLUME COLLECTED:

____

TOTAL WASTE VOLUME COLLECTED:

____

Form 2.4. SS and POC Sample Log

Station: ________________ Sampler: ____________________

Date	ISCO bottle number	Approximate Volume Collected	Approximate start time	Actual start time	Actual finish time	
	1					
	2					
	3					
	4					
	5					
	6					
	7					
	8					
	9					
	10					
	11					
	12					
	13					
	14					
	15					
	16					
	17					
	18					
	19					
	20					
	21					
	22					
	23					
	24					

Form 2.5. Sample Identification Log

Sample ID	Station	Date(s) Sampled	Time Sampled	Survey Number	XAD Column # (if applicable)	Type of Sample	Sampler Initials

Form 2.6. ISCO Sampler Program
Discrete samples at set time intervals

To program ISCO-2 to deliver two 1-Liter samples, starting every 30 minutes. As discussed previouly, a tee and valve will be placed after the pump tubing and before the distributor arm tubing, that will allow the filling rate to be controlled.

Turn on ISCO-2 (on/off key that has vertical line with circle around it), wait for main screen
Enter the numbers 6700.2 using keypad to ensure "Extended Programming" mode is set
 Screen will say: Please wait, Partition memory will last for 201 days
 Then: 6700 Sampler, Extended Programming, For help press ? At any time

Press down arrow to make "Other Functions" flash
Press Enter to select "Other Functions"
Highlight "Programming style", hit enter
Highlight "Normal", hit enter
Press arrows to make "Program" flash
Press enter to select "Program" to modify

Enter appropriate name using arrow and alphabet keys. Enter sets the letters (Enter 6-digit date, followed by station/river name first three letters: PAS-UP or PAS-DOWN, followed by "1" or "2" to indicate ISCO 1 or ISCO 2). Example: "062900PASSUP2" for an event starting on June 29, 2000 at the upstream site using ISCO 2.

Turn liquid detection off

Site description change? Yes, Enter same name as above, 062900ELI2

Select units for length, using the up arrow to highlight and enter to change the parameter as desired.
Highlight "ft", hit enter

Number of Bottles: 24
Bottle volume is: 1000 ml

Suction line length is: XXX
Enter "auto suction head"

0 rinse cycles
Retry up to 0 times when sampling

One part program

Uniform time paced
Time between sample events: Enter 1 hours, 0 minutes for small basins (Elizabeth, Rahway, and Hackensack) and 2 hour, 0 minutes for larger basins (Passaic and Raritan)

Form 2.6 . ISCO Sampler Program (continued)

Discrete samples at set time intervals

2 Bottles per sample event
Switch sets on: Number of samples
Switch sets every: 1 sample

Run continuously? Yes (must change bottles)

Do you want sample volumes dependent on flow? No

Sample volume? 1000 ml
Enable: None

Once enabled, stay enabled? No
Sample at disable? No
Sample at enable? No
Reset sample interval at enable? Yes
Continue countdown while disabled? Yes

Enter "Done" (No pauses)
No delay to start

Programming Complete Run This Program? No *(note: do not run program until all checks and settings are made and sample bottles have been installed).*

Return to main menu
Highlight and enter Other Functions
Go to Hardware:

- Use liquid detector? Yes
- Do you have a rain gage connected? No
- YSI 600? No
- Master/Slave? No
- Bottle Full Detect? Yes
- Continuous Backlight
- Event mark sent for: Every sample
- Event mark duration: Forward pumping
- During: Entire pump cycle
- Pre-sample purge ***(note: depends on tubing size and length at station):*** Use factory setting of "200 counts" and check end of line for bubbles during purge
- Post-sample purge (also depends on tubing size): Select "dependent on head"
- Set I/O: none, none, none
- Message: Analog output software has not been installed
- Periodic Serial Output? No (will change to Yes once Handar has been installed)

Form 2.6. ISCO Sampler Program (continued)

Discrete samples at set time intervals

At this point, press the red stop button to return to the main menu

Next: make sure the switch on the SPA 1156 box is in the "latch" position
Now it is necessary to take off the pump top of the ISCO to set the distributor arm to the correct setting. This is a very important step. For ISCO 1, the arm needs to be screwed all the way in. For ISCO 2, the arm needs to be screwed all the way out. If you look at the bottles and think about it and visualize it for a minute, this makes sense.
After calibrating sample volume and installing sample bottles, you can choose run program.

If at any time you wish to adjust parameters, grab a sample (1000 ml perhaps, to check is ISCO2 is still calibrated properly for volume delivered to bottles), or view the sampling report, press the red stop button once which temporarily interrupts the program, allowing you to make adjustments and then to resume the program. Pressing the red stop button twice, stops the program completely and must be restarted anew.

NOTE: Also useful after running program:
"View report" for both "sampling report" and "module"—you can see what ISCO did during the program
NOTE: If at any point you need to stop the program, press stop [or any another button, which turns the backlight on then press Stop again.
NOTE: If ISCO is not delivering desired volume to bottles, you may need to adjust these parameters:

- Suction head and length entered if not on continuous line and longer than 99 feet to intake
- Calibrate volume
- Rinse selected? If so, means purge, rinse, purge

NOTE: If ISCO shows note "pump cooldown" the sampler will automatically skip programmed samples for a mandatory ten minutes. This occurs because no liquid was detected at the detection point. Is the tubing out of the water?
NOTE: In order to reset ISCO to factory settings follow this procedure: Disconnect power source, both battery and battery plug for 30 seconds. Hold enter and red stop keys down for 5 seconds. Re-plug power source with two keys held down and continue to hold for 10 more seconds. This should restore any purge settings that may be out of whack due to adjustment on operator's part

Form 2.6. ISCO Sampler Program

Discrete samples at onset of ISCO 1 sampler, (set flow intervals)

To program ISCO-2 to deliver two 2-Liter samples every time ISCO 1 samples.
Turn on ISCO-2 (on/off key that has vertical line with circle around it), wait for main screen
Enter the numbers 6700.2 using keypad to ensure "Extended Programming" mode is set
    Screen will say: Please wait, Partition memory will last for 201 days
    Then: 6700 Sampler, Extended Programming, For help press ? At any time

Press down arrow to make "Other Functions" flash
Press Enter to select "Other Functions"
Highlight "Programming style", hit enter
Highlight "Normal", hit enter
Press arrows to make "Program" flash
Press enter to select "Program" to modify

Enter appropriate name using arrow and alphabet keys. Enter sets the letters (Enter 6 number date, followed by station/river name first three letters: RAR, ELI, RAH, HAC, PAS, followed by "1" or "2" to indicate ISCO 1 or ISCO 2). Example: "062900ELI2" for a storm starting on June 29, 2000 at Elizabeth using ISCO 2.

Site description change? Yes, Enter same name as above, 062900ELI2

Select units for length, using the up arrow to highlight and enter to change the parameter as desired.
Highlight "ft", hit enter

Number of Bottles: 24
Bottle volume is: 1000 ml

Suction line length is: *(See table below)*
Suction head: *(See table below)*

0 rinse cycles
Retry up to 0 times when sampling
One part program

Flow paced
Flow between sample events: 2 pulses
Sample at Start? No
1 Bottles per sample event

Form 2.6. ISCO Sampler Program (continued)

Discrete samples at onset of ISCO 1 sampler, (set flow intervals)

Switch sets on: Number of samples
Switch sets every 1 sample
Run continuously? Yes
Sample volume: 1000 ml

Enable: None
Once enabled, stay enabled? No
Sample at disable? No
Sample at enable? No
Reset sample interval at enable? No
Countdown while disabled? No
Next screen: Done, No pauses
No delay to start
Run this program now? No *(note: do not run program until all checks and settings are made and sample bottles have been installed).*

Return to main menu
Highlight and enter Other Functions
Go to Hardware:

Use liquid detector? Yes
Do you have a rain gage connected? No
YSI 600? No
Master/Slave? No
Bottle Full Detect? Yes
Continuous Backlight
Event mark sent for: Every sample
Event mark duration: While pumping
During: Forward pumping
Pre-sample purge ***(note: depends on tubing size and length at station):*** Use factory setting of "200 counts" and check end of line for bubbles during purge
Post-sample purge (also depends on tubing size): Select "dependent on head"
Set I/O: none, none, none
Message: Analog output software has not been installed
Periodic Serial Output? No (will change to Yes once Handar has been installed)

At this point, press the red stop button to return to the main menu

Form 2.6. ISCO Sampler Program cont.- Discrete samples at onset of ISCO 1 sampler, (set flow intervals)

Next: make sure the switch on the SPA 1156 box is in the "latch" position
Now it is necessary to take off the pump top of the ISCO to set the distributor arm to the correct setting. This is a very important step. For ISCO 1, the arm needs to be screwed all the way in. For ISCO 2, the arm needs to be screwed all the way out. If you look at the bottles and think about it and visualize it for a minute, this makes sense.

After installing sample bottles and calibrating the volume, you can now choose run program.
If at any time you wish to adjust parameters, grab a sample (1000 ml perhaps, to check is ISCO2 is still calibrated properly for volume delivered to bottles), or view the sampling report, press the red stop button once which temporarily interrupts the program, allowing you to make adjustments and then to resume the program. Pressing the red stop button twice, stops the program completely and must be restarted anew.

NOTE: Also useful after running program: "View report" for both "sampling report" —you can see what ISCO did during the program

NOTE: If at any point you need to stop the program, press stop [or any another button which turns the backlight on (kind of like a screen saver on a computer)] then press Stop again.

NOTE: If ISCO is not delivering desired volume to bottles, you may need to adjust these parameters:

- Suction head and length entered if not on continuous line and longer than 99 feet to intake
- Calibrate volume
- Rinse selected? If so, means purge, rinse, purge

NOTE: If ISCO shows note "pump cooldown" the sampler will automatically skip programmed samples for a mandatory ten minutes. This occurs because no liquid was detected at the detection point. Is the tubing out of the water? This is not a good thing.

NOTE: In order to reset ISCO to factory settings follow this procedure: Disconnect power source, both battery and battery plug for 30 seconds. Hold enter and red stop keys down for 5 seconds. Re-plug power source with two keys held down and continue to hold for 10 more seconds. This should restore any purge settings that may be out of whack due to adjustment on operator's part.

Form 2.7 USGS National Water Quality Analytic Request Form – See SOP #6

Form 2.8 USGS Kentucky District Sediment Laboratory Analysis Request Form - See SOP#6

USGS-SOP #3
Standard Procedures for Operating TOPS Samplers

1.0 General.
The operating and cleaning procedures that will be used in this study are detailed below and should be consulted for information regarding the operation and maintenance of this equipment. This document will provide the general reference to setting up, calibrating, and cleaning the TOPS samplers.

The TOPS consist of a peristaltic pump, flow-metering pump, two XAD columns, a canister glass fiber filter, and a flat glass fiber filter which will be used to collect samples for analysis of organic contaminants in dissolved and suspended sediment phases.

2.0 Cleaning.
Each TOPS used for collecting ambient water samples will be cleaned prior to use in the USGS District laboratory. The sampler will be removed along with its sampling inlet tube connections and returned to the laboratory. Empty, dedicated XAD columns, the glass-fiber filter (GFF) holder, the flat filter holder, the inlet sampling tube connectors, and the inlet/outlet bulkhead fitting caps will be washed in hot, soapy (liquid phosphate-free laboratory detergent) water, rinsed first in tap water, then rinsed with de-ionized water (DI) between use.

After reinstalling the dedicated cleaning-columns and GFF holder in the TOPS, 10 to 20 liters (L) of soapy tap water will be pumped through the TOPS sampler using a dedicated Teflon sampling line. This sampling line will be stored in a sealed plastic bag between uses. A new piece of Master-Flex tubing (Cole Palmer tubing IP 82 tubing, 18 inches long) will then be installed in the pump head following the manufacturer's instructions. Then two 20-L rinses of clean tap water will be pumped through the TOPS. The empty XAD columns will then be removed and the interior connection tube reconnected using the dedicated Teflon fittings. All metal parts will be rinsed in methanol, and 5 to 10 L of de-ionized water will be pumped through the TOPS and held inline. The mechanical tube fittings will then be checked for tightness, and the sample inlet and outlet ports will be tightly capped using the dedicated stainless steel caps. The TOPS case door will be closed and locked for transport.

The time, date, and initials of the TOPS cleaning will be recorded in the station logbook in Form 2.2 of USGS-SOP #2.

3.0 Description of XAD columns, Glass Fiber Filters, and Sample Bottles

3.1 XAD Columns.

The XAD columns are built from PFA Teflon parts purchased from Savillex Corporation, Minnetonka, Minnesota. Each column is built from the following parts:

1-Column body segment #504 125 ml volume
2-one-port transfer cap, 3/8-inch opening
2-Screen supports #504S
4-pieces of 150-micron Teflon cloth #1105- cut to fit into column body
2-3/8-inch ferrule nuts (used to connect columns to TOPS)
2-3/8” ferrule stop nits (used to seal inlet/outlets from columns).
Approximately 110 grams of washed and cleaned XAD-2 resin

One end-cap, two pieces of Teflon cloth, and one screen support are placed on the lower end of the column body. A unique identifier will be scratched into each column body. Resin is next poured as a slurry into the column, using lifts of approximately 1 cm at a time. The liquid is allowed to drain from the bottom of the column between each lift of resin. Once the column is filled within 1 cm of top edge of the column, two pieces of Teflon cloth, the screen support, and the top end cap are put in place. A small amount of ultra-clean laboratory de-ionized water is then squirted into the upper inlet, and the top and bottom end caps are put on place. The column is labeled with the following information:

Column and column lot numbers:
Date Packed:
Resin lot number:
Filled weight:
Direction of flow
SPIKED or NOT SPIKED
Initials of person who packed column.

Spiked columns will also have an arrow placed on the column showing the proper direction of water flow through the column.

3.2 - GFFs- Glass Fiber Canister Filters

For 4” canister filters:
Parker P/N K39R4G (0.5 µm), 4: long, no end treatment, 304 ss core.

Flat filters:
Whatman Grade GF/F glass microfibre filter paper
142 mm diameter Whatman #1825-142 (VWR #28497-907)—nominal pore size 0.7 µm

For filtration in lab for SOC/DOC:
Whatman 25 mm diameter GF/F glass microfibre filter paper
Whatman #1825-25 (VWR #28497-925)

Sediment from TOPS canister filter holder –1 cleaned baked amber wide mouth bottle equivalent to Eagle Pitcher Class #1 cleaned bottle #122-16A

4.0 Handling XAD Columns

The contracted analytical laboratory will be responsible for cleaning, refilling with new XAD-2 resin, blank checking, and where required, spiking with a mixture of ^{13}C-labeled compounds. The columns will be labeled, and sealed for transport and then shipped to the individual researchers. Each XAD column will be labeled with the date of cleaning, the type and amount of XAD resin contained, the type and amount of the spike that was added, and the batch number (if used). Each XAD column will be sealed with threaded Teflon plugs that are securely tightened. The XAD columns will be stored in a freezer when not in use, and will be transported in dedicated coolers that containing double-bagged ice. All XAD columns will be used within three months of the date of cleaning/preparation.

The XAD columns will be installed in the TOPS samplers immediately before being used for sampling. The column first in line to receive a water sample will be the spiked column and will be identified as column C-1, and the back-up column (furthest down the flow stream) will be identified as column C-2. The handler will wear disposable vinyl gloves when installing or removing XAD columns. The TOPS sampler, XAD columns, filter, and other parts will be set on a new sheet of clean plastic sheeting during preparation. Using an open-end or adjustable wrench of suitable size, the plugs sealing the internal tubing in the TOPS sampler will be removed. Then, beginning with the TOP of column C-1, the Teflon plug will be removed and the column connected to the TOPS sampler. The BOTTOM plug will then be removed and the XAD column connected to the TOPS sampler. The second XAD column C-2 will then be installed in a similar manner, connecting it first to C-1 and then to the TOPS tubing. The stainless steel and Teflon plugs will be stored in a dedicated, clean, plastic container until used to reseal the sampler and the XAD columns. Once the columns and the GFF filter have been installed, the location, date, time, and other such information required to identify the sample will be clearly marked on the column label using a permanent black marker.

Once a sample has been collected, the XAD columns will be removed in the order C-1 and then C-2. Beginning with the bottom connection of the column, the column will be disconnected using a dedicated open-end wrench and the Teflon plug immediately inserted into the column fitting and secured tightly to retain as much internal fluid as possible. Then the upper end of the column will be disconnected and sealed with a Teflon plug. The internal connections within the TOPS sampler will then be capped. The column labels will be marked with the date and time finished and the pair of columns wrapped individually in foil and then into a new, sealed, labeled, plastic bag and stored on ice until refrigerated or sent to the laboratory for analysis.

5.0 Handling GFF and In-Line Filters

5.1 GFF Installation

All GFF filters for the collection of suspended sediment will be cleaned by the manufacturer and then by the contract laboratory following laboratory procedures. Filters will be washed in clean methanol or hexane and baked at 400^{o} C overnight. The GFF filters will be wrapped in methanol or hexane washed aluminum foil and double-bagged in reusable plastic bags for shipping. During installation of the GFF filters, the handler will wear a new pair of disposable vinyl laboratory gloves. The GFF filter holder will be removed from the TOPS sampler using a dedicated open-end or socket wrench and any remaining rinse water discarded. The filter will then be removed from its aluminum foil or other shipping container using clean, methanol rinsed stainless steel forceps and placed in the holder. The filter will be checked to confirm a portion of the filter is above the edge of the holder, to provide a proper fit. The filter and holder will be immediately attached to the TOPS unit.

5.2 Changing and Removing GFFs

After sufficient sample has passed through the XAD columns, or in the event that the GFF becomes plugged with sediment, the GFF will be removed and stored for transport. The field person will don a new pair of clean, disposable laboratory gloves. The GFF filter holder will be unscrewed from the TOPS sampler, and then the filter will be removed using methanol rinsed, stainless steel forceps and wrapped in a new, clean 24-inch square sheet of aluminum foil. The foil-wrapped filter is then placed in a new, re-sealable plastic bag, sealed, and the outside of the bag labeled with the sample identification, date, time, and location. Proper field notation will be recorded on Form 8 of SOP#2 and on all labels. The filter bag is then bagged again. The combined filters will be stored on ice until frozen in the laboratory. Only the first canister filter and its corresponding flat filters will be sent in for analysis. The other filters will be stored in the freezer back in the district laboratory.

If filter has become clogged, installation of a new GFF filter is necessary using the above procedure. If sampling has ended sediment in the "dead-space" water of the canister filter holder (approximately 450 mL of fluid) will then be collected in the field by pouring the fluid into a wide-mouthed 500 mL bottle. The can will then be rinsed with de-ionized water. In the lab, the canister water will be filtered by pouring the water into the filter assembly that contains a pre-cleaned and baked GFF flat filter (nominal pore size of 0.7 µm) and diameter of 90 mm. The bottle will be rinsed with de-ionized water and the water will then be filtered under pressure of laboratory grade nitrogen gas. The filtered water will be discarded. Once all the water has been filtered, the filter paper will be removed and folded into quarters using stainless-steel forceps, wrapped in methanol-washed aluminum foil, and placed in the same reusable plastic bag package as its corresponding GFF sample and frozen.

5.3 In-line Filters

Filter pads in the inline filter holders (between the canister filter and the XAD columns) will be installed in the field before each event using methanol-rinsed metal forceps. The filter holders will be wrapped in aluminum foil and bagged in a re-sealable plastic bag for transport to the field. The filter holder will be installed in the TOPS using clean gloves using the bulls-eye bubble level to ensure filter is horizontal. After sampling or if the filter becomes clogged, inline filters are changed. The used filter is removed from the holder using methanol-rinsed tweezers, folded in quarters and placed in aluminum foil, folded and bagged with other filters. All times of use are recorded the field notebook. All inline filters will be sent in for analysis and become part of the filter pack that shares one ID number. A new inline filter is replaced using methanol-rinsed tweezers if sampling is to continue.

5.4 Evaluating Data Quality Problems from Sediment Breakthrough

There are no methods available to determine corruption by SS making its way through the flat filter and into the XAD. If in the unlikely event a flat filter is found to have broken or otherwise failed to trap sediment, it will be replaced and noted in the field notes (Note: all flat and GFF filters are combined together for extraction – for each sample collected.) Of the hundreds of samples collected in CARP there is only one recorded instance of flat filter failure. Analysis of the material collected on the flat filter of one sample in CARP showed it contained undetectable masses of chemicals – due in large part to the very small mass of sediment that is ultimately collected on each flat filter. (The GFF canister filter has a 10% breakthrough rating, but this outflow stream is split 1:10 before going through the XAD column. The net result is that even in high SS water, only an extremely small mass of sediment ultimately makes it to the flat filter.)

6.0 Procedure for operating a TOPS sampler.

The TOPS sampler will be used to collect a time composite sample of water and suspended sediment on traverses made during the three hours preceding high and low tide each day of dredging operations. Initial steps for collecting a TOPS sample involve making the sampling line connections, installing the TOPS filters (canister and inline filters), and checking the tubing connections for necessary tightness. The TOPS will be run manually by turning on the main power switch before each traverse is begun; this will also start the trace metal pump system (see USGS SOP-5).

The TOPS do not have any flow or pressure recording gages. The only values to calibrate and/or record are:

Flow rate of MF pump
FLOW rate of FMI pump
Total volume of water filtered
Total volume of water passed through XAD columns.

Flow rates are calibrated by recording the length of time 1L of water is collected from the two outflows (1 outflow for the GFF, and 1 outflow for the XAD). Flow rates will be calibrated before each day of sampling, and whenever time permits during the sampling. Generally, once set, the flow rates will not change during the day.

Volumes are determined by collecting water in a carboy, and measuring its volume with a graduated cylinder.

USGS-SOP #4
Procedures for Operating ISCO Samplers.

1.0 General.
ISCO #1 and ISCO #2collects discrete samples of river water at set time intervals, corresponding to the traversed of the TOPS boats. For the Lower Passaic Dredge Pilot Study, the samples will be collected 0.5 hours. The ISCOs-collects two1-liter samples at each sampling; one sample for suspended sediment and one for organic carbon and Br/Cl analysis. The samples are collected in dedicated pre-labeled bottles, and transferred to sample containers provided by the laboratory for analysis. The sample for organic carbon and Br/Cl analysis is filtered in the laboratory.

2.0 Cleaning.
Before each sampling event, the transfer lines and sampling bottles will be cleaned, and the pump and internal transfer tubing replaced. To clean the sampling lines, soapy water solution will be pumped through the line, followed by a minimum of 5 L of clean tap water. Bottles will be returned to the USGS district laboratory and cleaned using a hot soapy water solution and a soft bristle brush, followed by a clean tap water and a de-ionized water rinse.

2.0 Sample Bottles.
Discrete samples for TSS and organic carbon analysis will be collected in bottles equivalent to the ISCO 1-L wedge bottle, polypropylene bottle with polyethylene foam-lined lids, ISCO part number 68-3700-046

4.0 Program Verification.
Before each sampling event, the ISCO sampling program will be verified. The program is listed as Form 2.6 of SOP #2, reproduced below. A calibration program for the ISCO is listed in Table 1.3 of SOP #1. The program is not started until the bottles are put into the sampler.

5.0 Sampling.
ISCOs will be connected to the station sampling line using the dedicated transfer line. The 24 sampling bottles will be labeled, opened, and placed in the sampling rosette and the cover replaced immediately. The bottle lids will be stored in a Ziploc bag, placed in the dedicated plastic storage container, and left in the shed. Each bottle will be labeled using permanent marker and the label already on the bottle with the following information:

Stream______________
Location_____________
Date____________
Time_____________(of sampling—time needed from downloaded pgm.)
Sample No._________ (Ex. 2-2 for second set replaced and second bottle position)
Additional Data: (enter SS or Carbon here)

The ISCO program will then be started by pressing enter when “Start Program ____ now”, where ____ is the station name. If the sampler begins to sample and you are using the flow-integrated program rather than the set time interval, then immediately press the red stop button twice and enter when “ Stop Program” is flashing. The program should be verified again, and the sampler started once more.

The sampling crew should note the bottle numbers in the logbook.

6.0 Sample Processing.

The collected samples will be processed in the lab. One sample, labeled SS, will be sent in for Suspended Sediment analysis.

The second bottle, labeled Carbon, is for organic carbon and Br/Cl analysis. Upon the conclusion of the event, the appropriate samples will be chosen sent to the USGS district laboratory (or other designated laboratory), following their instructions. The POC sample will be filtered in the lab. The filter assembly consists of a Teflon filter holder, a 25 mm diameter GFF filter, and nitrogen gas as a form of positive pressure. The lab technician performing the filtering will wear laboratory gloves and use forceps to handle the filter and filter holder. The entire 1-L sample of water will be shaken vigorously, and a 125 ml aliquot will be filtered, using three filters. The filtered water will be collected and acidified with a 1.0 ml vial of 4.5 N (1:7) sulfuric acid (H_2SO_4) for DOC analysis. The filters will be removed, folded in half and placed in foil. The foil will be labeled with white tape with the information on the bottle label as well as the volume filtered. The foil packet will be placed in double Whirl-Pak bags and all Whirl-Paks placed in a Ziploc bag for shipment to the analytical lab for organic carbon and Br/Cl analysis. If necessary, the filtrate will be frozen and the filtered water stored in a dark refrigerator.

7.0 Data Retrieval.

Data from the ISCO-2 sampler will be retrieved using the RTD 581 device or a portable computer with the proper cables. The RTD number should be recorded on the proper form in the field notebook.

To use the RTD 581 device, plug the RTD into the instrument's interrogator connector port. The RTD device will work automatically. When working, the yellow light will blink to indicate power, then the green light will blink as the data reports are collected. The green light will remain solid when transfer is complete.

The red light reports two other RTD conditions: Memory Full, and Transfer Error. If the red light blinks, there has been a transfer error. Unplug the RTD, wait briefly, and repeat the transfer. If the transfer fails again, the ISCO should be returned to the USGS district laboratory for service.

Form 2.6. ISCO Sampler Program

Discrete samples at set time intervals

To program ISCO-2 to deliver two 1-L samples, starting every 30 minutes. As discussed previouly, a tee and valve will be placed after the pump tubing and before the distributor arm tubing, that will allow the filling rate to be controlled.

Turn on ISCO-2 (on/off key that has vertical line with circle around it), wait for main screen
Enter the numbers 6700.2 using keypad to ensure "Extended Programming" mode is set

Screen will say: Please wait, Partition memory will last for 201 days
Then: 6700 Sampler, Extended Programming, For help press ? At any time

Press down arrow to make "Other Functions" flash
Press Enter to select "Other Functions"
Highlight "Programming style", hit enter
Highlight "Normal", hit enter
Press arrows to make "Program" flash
Press enter to select "Program" to modify

Enter appropriate name using arrow and alphabet keys.

Turn liquid detection off

Site description change? Yes, Enter same name as above

Select units for length, using the up arrow to highlight and enter to change the parameter as desired.
Highlight "ft", hit enter

Number of Bottles: 24
Bottle volume is: 1000 ml

Suction line length is: XXX
Enter "auto suction head"

0 rinse cycles
Retry up to 0 times when sampling

One part program

Uniform time paced
Time between sample events: Enter value.

Form 2.6 . ISCO Sampler Program (continued)
Discrete samples at set time intervals

2 Bottles per sample event
Switch sets on: Number of samples

Switch sets every: 1 sample

Run continuously? Yes (must change bottles)

Do you want sample volumes dependent on flow? No

Sample volume? 1000 ml
Enable: None

Once enabled, stay enabled? No
Sample at disable? No
Sample at enable? No
Reset sample interval at enable? Yes
Continue countdown while disabled? Yes

Enter "Done" (No pauses)
No delay to start

Programming Complete Run This Program? No *(note: do not run program until all checks and settings are made and sample bottles have been installed).*

Return to main menu
Highlight and enter Other Functions
Go to Hardware:

Use liquid detector? Yes
Do you have a rain gage connected? No
YSI 600? No
Master/Slave? No
Bottle Full Detect? Yes
Continuous Backlight
Event mark sent for: Every sample
Event mark duration: Forward pumping
During: Entire pump cycle
Pre-sample purge ***(note: depends on tubing size and length at station):*** Use factory setting of "200 counts" and check end of line for bubbles during purge
Post-sample purge (also depends on tubing size): Select "dependent on head"
Set I/O: none, none, none
Message: Analog output software has not been installed
Periodic Serial Output? No (will change to Yes once Handar has been installed)

At this point, press the red stop button to return to the main menu

<u>Form 2.6. ISCO Sampler Program (continued)</u>
Discrete samples at set time intervals

Next: make sure the switch on the SPA 1156 box is in the "latch" position
Now it is necessary to take off the pump top of the ISCO to set the distributor arm to the correct setting. This is a very important step. For ISCO 1, the arm needs to be screwed all the way in. For ISCO 2, the arm needs to be screwed all the way out. If you look at the bottles and think about it and visualize it for a minute, this makes sense.
After calibrating sample volume and installing sample bottles, you can choose run program.

If at any time you wish to adjust parameters, grab a sample (1000 mL perhaps, to check is ISCO2 is still calibrated properly for volume delivered to bottles), or view the sampling report, press the red stop button once which temporarily interrupts the program, allowing you to make adjustments and then to resume the program. Pressing the red stop button twice stops the program completely and must be restarted.

NOTE: Also useful after running program:
"View report" for both "sampling report" and "module"—you can see what ISCO did during the program
NOTE: If at any point you need to stop the program, press stop [or any another button, which turns the backlight on then press Stop again.
NOTE: If ISCO is not delivering desired volume to bottles, you may need to adjust these parameters:

- Suction head and length entered if not on continuous line and longer than 99 feet to intake
- Calibrate volume
- Rinse selected? If so, means purge, rinse, purge

NOTE: If ISCO shows note "pump cooldown" the sampler will automatically skip programmed samples for a mandatory ten minutes. This occurs because no liquid was detected at the detection point. Is the tubing out of the water?

NOTE: In order to reset ISCO to factory settings follow this procedure: Disconnect power source, both battery and battery plug for 30 seconds. Hold enter and red stop keys down for 5 seconds. Re-plug power source with two keys held down and continue to hold for 10 more seconds. This should restore any purge settings that may be out of whack due to adjustment on operator's part.

<u>Form 2.6. ISCO Sampler Program</u>
Discrete samples at onset of ISCO 1 sampler, set flow intervals

To program ISCO-2 to deliver two 2-Liter samples every time ISCO 1 samples.
Turn on ISCO-2 (on/off key that has vertical line with circle around it), wait for main screen
Enter the numbers 6700.2 using keypad to ensure "Extended Programming" mode is set
    Screen will say: Please wait, Partition memory will last for 201 days
    Then: 6700 Sampler, Extended Programming, For help press ? At any time

Press down arrow to make "Other Functions" flash
Press Enter to select "Other Functions"
Highlight "Programming style", hit enter
Highlight "Normal", hit enter
Press arrows to make "Program" flash
Press enter to select "Program" to modify

Enter appropriate name using arrow and alphabet keys.

Site description change? Yes, Enter same name as above

Select units for length, using the up arrow to highlight and enter to change the parameter as desired.
Highlight "ft", hit enter

Number of Bottles: 24
Bottle volume is: 1000 ml

Suction line length is: *(See table below)*
Suction head: *(See table below)*

0 rinse cycles
Retry up to 0 times when sampling
One part program

Flow paced
Flow between sample events: 2 pulses
Sample at Start? No
1 Bottles per sample event

Switch sets on: Number of samples
Switch sets every 1 sample
Run continuously? Yes
Sample volume: 1000 ml

<u>Form 2.6. ISCO Sampler Program (continued)</u>
Discrete samples at onset of ISCO 1 sampler, set flow intervals

Enable: None
Once enabled, stay enabled? No
Sample at disable? No
Sample at enable? No
Reset sample interval at enable? No
Countdown while disabled? No
Next screen: Done, No pauses
No delay to start
Run this program now? No *(note: do not run program until all checks and settings are made and sample bottles have been installed).*

Return to main menu
Highlight and enter Other Functions
Go to Hardware:

> Use liquid detector? Yes
> Do you have a rain gage connected? No
> YSI 600? No
> Master/Slave? No
> Bottle Full Detect? Yes
> Continuous Backlight
> Event mark sent for: Every sample
> Event mark duration: While pumping
> During: Forward pumping
> Pre-sample purge ***(****note: depends on tubing size and length at station):* Use factory setting of "200 counts" and check end of line for bubbles during purge
> Post-sample purge (also depends on tubing size): Select "dependent on head"
> Set I/O: none, none, none
> Message: Analog output software has not been installed
> Periodic Serial Output? No (will change to Yes once Handar has been installed)
>
> At this point, press the red stop button to return to the main menu.

Next: make sure the switch on the SPA 1156 box is in the "latch" position.
Now it is necessary to take off the pump top of the ISCO to set the distributor arm to the correct setting. This is a very important step. For ISCO 1, the arm needs to be screwed all the way in. For ISCO 2, the arm needs to be screwed all the way out. If you look at the bottles and think about it and visualize it for a minute, this makes sense.

After installing sample bottles and calibrating the volume, you can now choose run program.

<u>Form 2.6. ISCO Sampler Program (continued)</u>
Discrete samples at onset of ISCO 1 sampler, set flow intervals

If at any time you wish to adjust parameters, grab a sample (1000 ml perhaps, to check is ISCO2 is still calibrated properly for volume delivered to bottles), or view the sampling report, press the red stop button once which temporarily interrupts the program, allowing you to make adjustments and then to resume the program. Pressing the red stop button twice, stops the program completely and must be restarted anew.

NOTE: Also useful after running program: "View report" for both "sampling report" — you can see what ISCO did during the program

NOTE: If at any point you need to stop the program, press stop [or any another button which turns the backlight on (kind of like a screen saver on a computer)] then press Stop again.

NOTE: If ISCO is not delivering desired volume to bottles, you may need to adjust these parameters:

- Suction head and length entered if not on continuous line and longer than 99 ft to intake
- Calibrate volume
- Rinse selected? If so, means purge, rinse, purge

NOTE: If ISCO shows note "pump cooldown" the sampler will automatically skip programmed samples for a mandatory ten minutes. This occurs because no liquid was detected at the detection point. Is the tubing out of the water? This is not a good thing.

NOTE: In order to reset ISCO to factory settings follow this procedure: Disconnect power source, both battery and battery plug for 30 seconds. Hold enter and red stop keys down for 5 seconds. Re-plug power source with two keys held down and continue to hold for 10 more seconds. This should restore any purge settings that may be out of whack due to adjustment on operator's part.

USGS-SOP #5.
Procedures for collecting and processing temporally integrated samples for trace metals.

1.0 General Summary.

During each sampling event, temporally integrated samples will be collected for dissolved and total (whole water) trace metal concentrations. These samples are to be collected during the traverses completed during the three hours preceding high and low tides, respectively (seven traverses each tide cycle). This SOP describes the procedures and equipment used for collecting and processing the samples.

Sampling for trace metals will be done using a peristaltic pump connected to a dedicated sampling line. After the pump head, the tubing will; be split into four lines, each emptying into four bottles. The general schematic for this process is shown on QAPP Figure B-6. The contact laboratory will supply dedicated pre-cleaned tubing for use in the pump, the filters, and bottles. Four samples will be collected each tide cycle that is sampled, consisting of two 2.5-L unfiltered samples, and two 2.5-L filtered samples. (Note: the fourth bottle is a spare; as both the filtered and unfiltered Hg will be submitted in a single bottle for aliquoting and filtration at the trace mercury laboratory. The fourth bottle position may be utilized to collect a field duplicate for trace Hg, filtered metals, or total metals.) The 0.45 micron (µm) capsule filters, supplied by the contract laboratory, will be used to filter the samples in the filed as they are collected. The samples will be collected, labeled, and field data recorded in the station logbook.

The peristaltic pump needs no real calibration after initial set up- they only need to pump approximately the same volume of water each traverse. The pumps will be set and checked each day in the morning; new pump tubing will be used for each sample.

2.0 Cleaning of Sampling and Filtering Equipment.

The pump tubing, transfer lines, filters, and bottles will be supplied precleaned by the analytical laboratory. No cleaning of these supplies will be done by the field sampling crew. The inlet tubing for the metal sampling, leading from the weighted "fish" to the peristaltic pump, will be cleaned before each use (before each day of use). If necessary, the inlet end of the tubing will be trimmed off to remove any heavily soiled tubing. The tubing will be cleaned by pumping hot soapy tap water through the tubing, followed by a tap water rinse, rinsed with de-ionized water. Then a 10 percent HCl solution will be pumped through the tubing, followed by a rinse with laboratory de-ionized water. The tubing will be stored in a clean plastic bag and will be reinstalled in the morning before each use. All fittings will be composed of Teflon, and will be cleaned by washing in hot soapy tap water, rinsing in de-ionized water, soaking in 10 percent HCl solution, rinsing in laboratory de-ionized water, and sealing in plastic bags for transport to the field.

2.1 Setup

The inlet tubing will be connected to the weighted fish and run up the weighted line and into the boat, securing with cable ties. The tubing will be connected to the supplied pump tubing, put through the sampling head, brought out of the head, and split into four lines as shown in QAPP Figure B-6. The filter and tubing will be held in place using a ring-stand and clamp system that will be clamped to the gunnel of the watercraft. All connections will be made using Teflon barbed fittings with plastic hose clamps.

2.2 Sample Bottles

Samples for metal analysis will be collected in 2.5-L, pre-cleaned (acid washed for trace mercury) bottles supplied by the contract laboratory. Sample bottles are double-bagged and the sample bottle is not handled by the field sampling team.

3.0 Procedure.

Two metal samples will be collected – one for trace Hg analysis and the second for a suite of metals. Hg analysis will be performed using method EPA 1631E (by a contract laboratory; STL-North Canton), the metal suite will be analyzed the USEPA DESA laboratory as detailed in the QAPP. These analyses will be performed on unfiltered and filtered samples collected concurrently from the same inlet line. The difference between concentrations in unfiltered and filtered samples will be assumed to represent the concentration in the suspended sediment phase.

3.1 Sample Collection Procedure

1. For both the Hg and the TAL metal samples, one unfiltered sample will be collected over seven traverses (in some cases, fewer traverses may be conducted, as noted in the Work Plan). A field-filtered sample will also be collected for TAL metals (the trace Hg sample filtration will be conducted at the laboratory.) These traverses will be conducted beginning each ½ hour up to maximum high and minimum low tide.

2. Each of the four "sample" types will be approximately 1.960 Liters in volume. Each traverse will collect 280 mL of water. The exact volume is not critical, so long as the 2L volume is not exceeded. The pumping rate will be set depending upon the traverse speed that can be maintained. It is not critical to collect the exact same volume in each sample because (1) dissolved concentrations are not a function of sample volume, and (2) SS bound concentrations are based upon the difference between dissolved and unfiltered samples. Every effort will be made to adjust the pump rates to collect approximately the same volume each traverse and not to exceed the 1.96L target.

3. QAPP Figures B-5 and B-6 shows a schematic of the sampling train.

4. The sampling will be conducted as follows:

4.1 The operator will don new clean gloves ("non-talc"), and lay the bagged sampling tubes and filters on clean plastic in the sheltered sampling area on the boat.

4.2 Pump "calibration" tubing, Y connectors, and filters will be installed in the pumps. The pumps will be started and the flow rates will be checked and recorded. Once adjusted, the calibration tubing, Y's and filters will be removed.

4.3 New, clean pump tubing, Y's and filters will be installed in the pumps, and capped bottles will be placed in bottle racks.

4.4 One field blank will be collected from the unfiltered and one from the filtered line. These samples are prepared by placing the inlet line end of the pump tubing directly into a new bottle of laboratory water provided by the contract lab. One line from each TEE will be clamped off (only one filtered and one unfiltered field blank will be collected). Once the lines have filled and the air expelled, the outlet end will be placed into a sample bottle and at least 1.75L of water collected. The field blank bottles will be resealed in their double bag, and labeled on the outside of the inner bag.

4.5 The inlet end of the pump tubing will be connected to the outflow end of the large peristaltic pump tubing, and the inlet end of the large peristaltic pump tubing will be connected to the sampling line. The outlet end of the small peristaltic pump tubing and the outlet end of the cartridge filters will be placed over a waste bottle. The large pump will be started, and once river water has been brought up the line, the two small pumps will be started and the outlet tubing and cartridge filters will be allowed to fill. All air will be removed from the cartridge filters. Once filled, the two small pumps will be stopped. The ends of the small pump tubing and cartridge filters will be encased in clean plastic bags held on with cable ties until the traverses for sampling commence. The large peristaltic pump can be left running before and between each traverse.

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4.6 Immediately before the water leaves for the each traverse, the small peristaltic pumps will be turned on and the sample line and filters will be flushed with river water. Once the lines have been sufficiently flushed, a signal will be sent to the boat operator and the traverse will begin. The small peristaltic pumps will be shut off when the boat returns to the 2m depth buoy at the south shore line (the starting shore). The small peristaltic pumps can be shut off upon completion of each traverse, while the large pump can be left running.

4.7 Immediately after the last traverse is completed, the pumps can be shut off, the tubing removed from the sample bottles, and the sample bottles can be capped and sealed in double zip-lock bags. The sample label will be placed on the inner bag, and the sealed samples placed immediately on ice.

3.2 Equipment Blanks

Before the first day of sampling, a field equipment blank will be collected. This blank will be produced by pumping laboratory-supplied de-ionized water (mercury-free water for trace Hg) through the pump tubing and the sampling tubing. An unfiltered blank sample of about 1 L sample volume will be produced. Filtered blanks will be made by first holding the filter vertically until the cartridge filter fills with water. During the preparation of the field blanks the pumping rate will be adjusted to 25 mL/minute. The blanks will be capped, sealed in two plastic zip lock bags, and the outer bag labeled with date, time, and sample location. After the blanks have been produced, the peristaltic pump tubing will be connected to the inlet sampling line.

3.3 TOPS Sampling.

The trace metal samples will be collected only during the traverses in which the TOPS sampler is operated (three hours before high and low tide); see work plan for specific times and dates. The peristaltic pump will be plugged into the auxiliary power outlet on the TOPS. The pump will be left turned on so it operates only when the TOPS is operating.

Immediately before the first TOPS traverse is begun, the TOPS sampler will be run to fill the tubing and canister filter with river water. At the same time, the metal sampling pump will be run and its lines filled with river water. The capsule filters will be inverted and allowed to fill with river water, and the pump rate will be checked. Once the system is charged, the pump will be stopped, the lines placed into the respective bottles, and a signal will be given to the boat operator to begin the traverse. At that time, the pump will be restarted (along with the TOPS sampler) and the composite sample prepared during the traverse. Upon completion of the traverse, the TOPS will be stopped (thereby stopping the metal sampling pump). The system will then be ready for the next tide cycle by replacing the pump tubing, the system tubing, filters, and all fittings.

Upon completion of the seven (nominal) traverses comprising a 3-hour sampling event (note that in some cases fewer traverses will comprise a sampling event), the pumps will be stopped, and the bottles capped. No preservatives will be added to the samples in the field. The sample bottles will be double-bagged, and the sample label is placed between the outer and inner bag. The label will include station ID, date, and time. Samples will be placed on ice in coolers, the chain-of-custody forms will be completed, and the coolers shipped to the contract laboratory as soon as possible (on the day of collection, if possible) for analysis.

USGS-SOP #6
Preparation of TSS and Organic Carbon Samples, and TOPS Canister Filter Water.

1.0 General.

Two types of samples may require filtering at the end of this work; the residual water and sediment in the canister filter housing of the TOPS, and the POC samples require filtering for submission to the USEPA DESA laboratory. Although no special handling or preparatory work is required for the suspended sediment samples, these samples will need to be secured and shipped for analysis.

2.0 Filtration of sediment collected from the TOPS canister filter

Sediment collected in the TOPS filter canister is transferred in the field to a pre-cleaned 500-mL wide mouth bottle, and transported chilled, and stored in the refrigerator in the lab. The sediment is captured through filtration for analysis using the following procedure.

Use vinyl gloves for all procedures and handling all equipment and supplies. Pre-wash filter holders and tools with phosphate-free laboratory detergent, rinse with organic-free de-ionized water, apply methanol, and follow with another rinse of organic-free de-ionized water. Filter holders and associated tools will be kept covered in aluminum foil and in Ziploc bags.

A large pressurized stainless steel filter holder is available at the district laboratory for filtering the GFF filter water from each TOPS canister. This filter holder will be washed in hot soapy water, rinsed in tap water and then de-ionized water, rinsed with clean methanol, and allowed to dry between use. The filter holder will be stored foil and plastic bags in a dedicated storage container.

Place 142 mm diameter GFF filter on filter holder assembly, pre-rinse the filter paper with de-ionized water, and assemble reservoir following manufacturer's instructions. Shake the sample bottle vigorously to re-suspend sediment. If necessary, remove 5 to 10 mL of sample to allow for small headspace in bottle before shaking. Transfer sample to the filter reservoir, and rinse the sample bottle with sufficient de-ionized water, transferring this rinse to the filter reservoir. Place filter assembly cap on, connect filter to nitrogen tank, and apply no more than 10 psi of pressure. Filtered water can be discarded. Once sample has been filtered, release pressure, open filter assembly, rinse reservoir with sufficient volume of de-ionized water, filter again, and discard filtrate. Release pressure, open filter assembly. Using pre-cleaned forceps, remove filter paper and place on a new piece of aluminum foil. Fold filter into quarters with forceps and fold foil around filter making a sealed packet. Place foil packet in its corresponding sample filter pack (the labeled double zip-lock bag containing the TOPS canister filter and all flat filters used in the field during the collection of the sediment sample). Rinse filter housing in phosphate-free laboratory detergent, de-ionized water, and methanol between samples.

TOPS canister samples are collected for each TOPS sample, capturing the water and sediment that remains in the canister filter housing after the canister filter is removed. In the field, this material is poured into 500 mL wide mouth bottles and the canister filter housing is rinsed with lab water. The bottle is sealed and labeled and returned to the lab with its associated canister filter sample. In the lab, the 500 mL bottle is opened and the material poured into a 145mm SS filter housing, sealed, and pressure filtered (using N_2 gas). The filter housing is rinsed, the bottle is rinsed and filtered. Upon completion, the filter paper is folded and placed in aluminum foil and added to the GFF filter bag (which contains the GFF canister filter and flat filters). These filters constitute one sediment filter.

The method blank for the TOPS canister filter is included in the TOPS equipment blank. One TOPS equipment blank will be prepared for each TOPS sampler used in the study. No individual result will be obtained for the TOPS canister filter – it is included in the analysis of the canister filter paper, the flat filter paper, and the canister flat filter.

In summary – 1 TOPS sediment samples includes:
One GFF canister filter
One or more GFF flat filters
One GFF "canister" flat filter contain residual sediment from the canister filter

POC samples are three replicates of filtered material retained by passing 60 to125 mL of sample through 25-mm diameter glass fiber filters. These three replicates are sealed in foil and sent as one labeled sample (unique identifier, time and date, and sample boat ID). The results obtained from the laboratory is the average of the ignition of each of these three filter papers.

3.0 Discrete Samples for Particulate Organic Carbon (POC) Analysis.

As necessary, the discrete POC samples that are collected in the second ISCO sampler will be prepared by filtering and appropriate volume (125 mL) through 25mm diameter pre-baked glass fiber filters. Filter pads will be supplied by the National Water Quality Laboratory in sealed boxes (or equivalent obtained from a commercial vendor). Three 60-mL aliquots of each sample will be filtered. The three filters will be wrapped in pre-baked aluminum foil squares, double bagged, and labeled. Filtered samples will be stored frozen until all samples have been processed. The samples will be shipped on ice to the contract laboratory for analysis.

The filter holders used for POC sample filtering will be cleaned by washing in hot phosphate-free soap solution, rinsing in organic-free de-ionized water, wrapping in aluminum foil, and packing in double Ziploc bags for storage until use. New, pre-cleaned, pre-baked glass fiber filters will be used for collecting POC and will require no further cleaning before use. Filters, bottles, and other equipment are to be kept away from methanol and other organic solvents. Between samples, the filtering equipment will be cleaned in soapy water and rinsed in de-ionized water.

3.1 Filtration Equipment Needed for POC analysis

Teflon pressure filter – Savillex model DOC25 – 25mm diameter filter holder, or 25mm glass
25-mm diameter glass microfiber filter – Whatman 25 mm diameter Whatman #1825-025 (VWR # 28497-925) or equivalent
Stainless steel 142mm diameter pressure filter and reservoir
142 mm diameter glass microfiber filters Whatman #1825-142 (VWR # 28497-907)
Laboratory grade compressed nitrogen, with tank regulator and copper tubing transfer line
Stainless-steel forceps
Small plastic petri dishes with covers – 150-mm diameter x 25-mm deep, pre-sterilized
Glass graduated cylinder

Squirt bottle with organic-free water
Squirt bottle with clean methanol
Labeling tape-white
Clear masking tape
Electrical tape
1.0 ml vials of 4.5 N (1:7) sulfuric acid (H_2SO_4) [Eagle-Picher Part #05285 UN 1830]
Permanent fine-point marking pen
Vinyl laboratory gloves
Whirl-Packs and Ziploc bags
Aluminum foil
Neon-colored label dots for sample identification

3.2 Filtration of POC samples

Filtering is to be done within two weeks of collection of samples for POC analysis, following the USGS NAQWA procedures outlined in NWQL Technical Memorandum 2000.04.

Use vinyl gloves for all procedures and handling of all equipment and samples. Pre-wash filter holders and tools with phosphate-free detergent, rinse with organic-free de-ionized water. Keep filter holders and tools covered in aluminum foil.

Place 25-mm diameter glass fiber filter on filter holder assembly and connect to reservoir following manufacturer's instruction. Pre-rinse the filter with 5 to10 mL of de-ionized water. Shake sample bottle vigorously to re-suspend sediment. If necessary, BEFORE SHAKING, remove 5 to 10 mL of sample to allow for small headspace in bottle. Rinse graduated cylinder with a small portion of sample, discard, then pour 125 mL of sample into graduated cylinder. Transfer this to the filter assembly, rinse graduated cylinder with 5 to 10 mL of de-ionized water, and transfer to the filter assembly. Place filter assembly cap on, connect to nitrogen tank, and apply no more than 10 psi of pressure. Collect filtrate in pre-baked 125 mL amber glass bottle with Teflon lined cap. Acidify sample for DOC analysis with a 1.0 ml vial of 4.5 N (1:7) sulfuric acid (H_2SO_4). (Do not preserve samples for Br/Cl analysis.) Cap and seal lid with electrical tape.

Once sample is completely filtered, release pressure, open filter assembly cap, rinse the sides of the reservoir with sufficient volume of de-ionized water, close filter assembly and filter, discarding the filtrate. Using stainless steel forceps, remove the filter from the filter assembly; fold in half, place in foil and fold foil into a packet. Repeat this procedure two more times so as to obtain three 25-mm GFF filters for POC analysis. Place all three filters in one foil packet and place foil packet into two Whirl-Paks and label the inner Whirl-Pak with a piece of white tape containing the following information:

River name/station number and ISCO bottle number (if applies)
Date of collection
Time of collection
Volume filtered (e.g., 3 x 125 mL filtered)
SC 2631–SOC
T. Wilson, N.J.
4891-12300

Combine Whirl-Paks in a zip-lock bag for shipping.

Label all filtered, acidified water samples for dissolved organic carbon (and Br/Cl; no preservative for Br/Cl) analysis as follows:

River name/station number and ISCO bottle number (if applies)
Date of collection
Time of collection
LC 2613–DOC
T. Wilson, N.J.
4891-12300

Use white labeling tape and a Sharpie or similar pen for the label and put clear masking tape over the white label so that it will stay on. Tape lid of amber bottle securely with electrical tape.

Keep all bottles and filters in the refrigerator until they are shipped. Rinse filter assembly with phosphate-free detergent and de-ionized water between samples.

In addition, a neon-colored label dot with a numerical identification needs to be placed on:
(1) DOC sample bottle top
(2) corresponding SOC filter label,
(3) upper left hand corner of Analytical Services Request or COC Form, and
(4) first left hand column marked “Number” on the chain-of-custody form.

3.3 Laboratory (Method) Blanks

One method blank will be prepared for each 10 to 20 samples. Method blanks are produced by passing 125mL of laboratory water through each of three 25-mm GFF filters, using the same procedures used for samples. The three filter pads are wrapped in foil and treated as one sample by the lab. The resulting POC will be the average of the carbon released upon ignition of the three individual filter pads. The filtered water is used for the blank for the Br/Cl and DOC analysis, as needed.

4.0 Discrete Samples for Total Suspended Solids Analysis

Concurrent with the automatic collection of the samples for organic carbon analysis (described in section 3.0) will be samples for TSS (suspended sediment) analysis. These samples are collected by ISCO-1 in 1000-mL wedge shaped poly bottles identical to the ones described earlier. These bottles are changed daily during the visits by field crew, with the caps being taped in place for transport. The bottles are pre-cleaned and have yellow labels attached to them before use. Since the bottles and caps are pre-weighed by the lab, it is imperative that the caps are also numbered and kept with the corresponding sample bottles. Caps are kept in a plastic zip-lock bag stored within the ISCO sampling chamber. The sample identifications and bottle numbers are recorded on the labels when the bottles are placed in the sampler, and are also recorded in the station logbook.

Once the samples have been collected and returned to the laboratory, the samples that will be analyzed are selected and prepared for shipment. Label water level with a grease pencil. Pack the selected ISCO bottles in a cooler (24 maximum per each cooler). Fill out a chain-of-custody form which can serve as the sample shipment log form. Make copies of the sample shipment log form and put them in the respective file folder. Place the original form(s), a completed copy of the chain-of-custody form (Form #5 or equivalent) and a “return address label” in a Ziploc plastic bag and tape it to the inside of the cooler lid.

Methods blanks for SS are not prepared in the field. They are prepared in the laboratory according to the lab QAPP – typically 1method blank per 10 to 20 samples. These are treated identically to the samples.