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PHASE 2B
SAMPLING AND ANALYSIS/QUALITY ASSURANCE PROJECT PLAN

VOLUME 1: FLOW-AVERAGED WATER-COLUMN SAMPLING

HUDSON RIVER PCB REASSESSMENT RI/FS

USEPA WORK ASSIGNMENT NO. 013-2N84

REVISION 1

March 1993

Prepared for

USEPA Region II

Alternative Remedial Contracting Strategy (ARCS)

for

Hazardous Waste Remedial Services

Prepared by

TAMS CONSULTANTS, INC.
and
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Hudson River PCB Reassessment
RI/FS Phase 2B SAP/QAPjP - Volume 1
March 1993

Revision 1

	Date
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* These appendices have been previously submitted to USEPA for review (Phase 2B, Volume 2 SAP/QAPjP, Revision 0, dated February 25, 1993) and are therefore not included here.

** The appendices are essentially identical to these submitted previously with the Phase 2A SAP/QAPjP (Revision 2, May 29, 1992). However, minor modifications (corrections of typographical errors and rephrasing of text) have been made along with some changes to QC criteria; therefore, these appendices are included.

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3 Project Description

In accordance with the Scope of Work for the Hudson River PCB Reassessment RI/FS (December 1990) and the Final Phase 2 Work Plan and Sampling Plan (September 1992), Phase 2 of the Reassessment involves field sampling to further characterize and analyze site conditions at the Hudson River PCB Superfund Site. The Phase 2A Sampling and Analysis Plan/Quality Assurance Project Plan (Revision 2 dated May 29, 1992) described four sampling activities, including confirmatory/geophysical sediment sampling and high-resolution sediment coring (both of which have been completed) and water-column transect and water-column PCB equilibration studies (both of which are ongoing).

Phase 2B sampling will include five components, as described in the Final Phase 2 Work Plan and Sampling Plan: flow-averaged water-column sampling; low-resolution coring of Upper Hudson River sediments; analysis of archived water column and sediment samples on a PCB congener-specific basis; sediment critical shear stress analysis; and ecological sampling. An additional sampling program involving dye releases into the Hudson in order to establish the time-of-travel between sampling points may also be implemented in Phase 2B. Only the flow-averaged water-column sampling is described in this report: **Volume 1 of the Phase 2B Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPjP).** Additional volumes of the Phase 2B SAP/QAPjP containing details of the remaining field programs will be submitted separately.

3.1 Background

3.1.1 Site Description

The Hudson River PCB Superfund site encompasses the Hudson River from Hudson Falls (River Mile [RM] 198) to the Battery in New York Harbor, a stretch of nearly 200 river miles. Because of their different physical and hydrologic regimes, the Upper Hudson 40 mile stretch, from Hudson Falls to Federal Dam (RM 155), is distinguished from the Lower Hudson stretch, from Federal Dam to the Battery. The part of the Upper Hudson from Bakers Falls to the Sherman Island Dam (designated as study Area A on Figure 3-1) is not part of the Hudson River PCB Superfund site, but serves as a background or control area. At this time, potential remedies for PCBs in sediments at the site are limited to river bottom sediments of the Upper Hudson. However, investigations into PCBs in the Lower Hudson are an integral component of understanding the past

and present migration of PCBs, dissolved or bound to suspended matter in water, from the Upper Hudson to the Lower Hudson.

3.1.2 Site History

During an approximately 30 year period ending in 1977, two General Electric (GE) facilities, one in Fort Edward and the other in Hudson Falls, NY, used PCBs in the manufacture of electrical capacitors. Various sources have estimated that between 209,000 and 1,300,000 pounds of PCBs were discharged between 1957 and 1975 from these two GE facilities. Discharges resulted from washing PCB-containing capacitors and minor spills.

The PCBs discharged to the river tended to adhere to sediments and subsequently accumulated downstream with the sediments as they settled in the impounded pool behind the former Fort Edward Dam. Because of its deteriorating condition, the dam was removed in 1973. During subsequent spring floods, PCB-contaminated sediments were scoured and released downstream. Exposed sediments from the former pool behind the dam, called the "remnant deposits," have been the subject of several remedial efforts.

Investigations at the site began after PCBs were reported in fish caught in the Upper and Lower Hudson in the early 1970s. In 1971, New York State Department of Environmental Conservation (NYSDEC) added PCBs to their statewide analyses of pesticide residues in fish, although no results were released publicly until 1975. After USEPA investigations in 1974 of PCB contamination in the Fort Edward area, NYSDEC intensified its PCB sampling program. In 1976, following the 1975-76 fish monitoring effort, NYSDEC banned all fishing in the Upper Hudson river from Albany north to Fort Edward due to the high levels of PCBs in fish. Commercial fishing for striped bass in the Lower Hudson was also closed at the same time. Both bans remain in effect. In addition to the ban on striped bass, New York has banned the sale of other Hudson River fish, including American eel, white perch, carp, goldfish, brown bullhead, pumpkinseed sunfish, white catfish, and black crappie.

USEPA under the National Contingency Plan (NCP) and Comprehensive Environmental Response Compensation and Liability Act (CERCLA), or Superfund, process performed a Feasibility Study in 1984 and issued a Record of Decision (ROD) for the site in 1984. The ROD called for: 1) an interim No Action alternative for river sediments; 2) in-place containment, capping, and monitoring of the remnant deposit

sediments; and 3) a treatability study to evaluate the effectiveness of the Waterford Treatment Plant in removing PCBs from the river water. Since the signing of the ROD, the planned remedial efforts of the remnant deposits have been completed. The Waterford Treatment Plant treatability study concluded that the water supplied for drinking water meets Federal and State standards.

In 1989, USEPA announced that the No Action alternative for Upper Hudson river sediments would be reassessed, and in 1990 issued a Scope of Work outlining a three phased reassessment:

Phase 1	•	Preliminary Reassessment or Interim Site Characterization and Evaluations
Phase 2	•	Further Sampling and Analysis
Phase 3	•	Feasibility Study

The Phase 1 Report (Interim Characterization and Evaluation-Review Copy) was issued in August 1991. In order to complete the entire investigation in a timely manner, an initial sampling program, called Phase 2A, was proposed and implemented by USEPA in November, 1991. The Phase 2A Sampling Plan-Revision 2 (TAMS, May 29, 1992) outlines the sampling tasks for Phase 2A. The complete Phase 2 Work Plan was issued in September, 1992. This Phase 2B (Volume 1) SAP/QAPjP covers the first of five site investigation tasks to be conducted under Phase 2B.

3.2 Project Objectives for Flow-Averaged Water-Column Sampling Study

The Phase 2B analytical program for the Hudson River can be separated into five basic studies. Each study is designed to meet a specific project objective. This volume (Volume 1) of the Phase 2B SAP/QAPjP details the flow-averaged water-column sampling program. This program is designed to determine relatively long-term averages of water-column conditions in the Upper Hudson by compositing samples at a specific location over a fifteen day period to account for variations in flow (discharge), suspended matter load, sediment scour, etc.

The purpose of this sampling is to define better the net PCB loads to the Upper Hudson that enter the river as it travels through the remnant-deposit area above Fort Edward and through the Thompson Island Pool downstream to the Thompson Island Dam. The river sections under study represent regions of known or suspected historic (and possibly current) PCB input to the river. Mean differences in PCB levels between sampling stations represent net changes in PCB load resulting from a PCB source, a PCB sink or dilution in the intervening river section. Actual loadings will be calculated using USGS flow data and the measured PCB values.

The Phase 2B flow-averaged water-column study is intended to address several issues concerning riverine PCB contamination, including:

- the source or sources of PCBs at Ft. Edward which, on an annual basis, appears to be a current major source to the Upper Hudson, as suggested by the Phase 1 Report;
- temporal variations in the source of PCBs in the Upper Hudson, and;
- the factors governing PCB transport and water column concentrations such as seasonal or flow variations.

While it is not anticipated that the flow-averaged water-column sampling and analytical program described in this SAP/QAPjP can resolve all these issues, it is expected to clarify many of them. The results of other Phase 2A and 2B programs, mainly the high resolution coring, low resolution coring, and water transect programs, will also help to clarify these issues. The success of the Phase 2B effort is dependent upon both the quality of the measurements made and the actual results obtained. The individual analyses scheduled for the flow-averaged water column study are discussed below within the context of meeting the program data quality objectives. The analyses will be performed by contract laboratories and Rensselaer Polytechnic Institute (RPI).

The measurements produced by the flow-averaged sampling program represent a perspective on river conditions midway between the instantaneous conditions determined by the water-column transect sampling currently underway and the long-term average water-column conditions determined by the high-resolution sediment coring program completed in November, 1992. The ultimate goal of this program is to provide a measure of mean total PCB transport. Since collection and analysis of daily large volume samples for PCB

3.4.2 Dissolved Organic Carbon

Dissolved organic carbon (DOC) is defined as the total organic carbon content of a filtered water sample. DOC has been shown to affect dissolved phase/suspended matter phase partitioning of PCBs. Presumably, higher levels of dissolved organic carbon result in greater micelle and colloid formation. In turn, these micelles provide a greater capacity for the support of PCBs in a "dissolved" form.

The persulfate oxidation method represents the continuation of an existing database of DOC measurements, a data set that has been correlated with many historic water-column PCB analyses. DOC analyses will be conducted on flow-averaged composite samples.

3.4.3 Total Suspended Solids (TSS) and Weight-Loss-On-Ignition (WLOI)

These analyses will be conducted on the suspended matter (non-filterable residue) portion of the composited and filtered water sample. The TSS/WLOI analyses will determine the amount of solid material per volume of water sample. In addition, the WLOI analysis will provide an estimate of the organic carbon content of the suspended matter by combustion of the non-filterable suspended solids retained by the glass-fiber filter. TSS analyses will also be conducted on separate discrete grab samples collected during the flow-averaged composite sample collection. WLOI analyses will be conducted only on discrete grab samples in which significant TSS concentrations (25 mg or more) are detected.

3.4.4 Temperature, pH, Conductivity, and Dissolved Oxygen

Temperature, pH, and conductivity will be measured as standard indicators of water quality conditions. Dissolved oxygen will be measured as a general indicator of water quality conditions and as a crude measure of gas exchange capabilities in various reaches of the river. It may prove most useful in examining the effect of dams and spillways in the Upper Hudson on gas exchange. These features may have important implications for loss of water-borne PCBs to the atmosphere, particularly the lightest congeners.

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analysis is not practical, this flow-averaged sampling program has been designed to make efficient use of available project resources by collection and analysis of composite samples collected over time (15-day periods). Since an accurate suspended matter/dissolved phase distribution of PCB congeners is not of critical importance for this flow-averaged program, PCB samples will not be filtered on the day of collection. (See Section 6 for sampling procedures.) The alternative means of obtaining similar information is to collect and analyze a large number of samples and arithmetically average (or composite) the results. Discrete grab samples (in addition to the flow-averaged composites) will be collected and analyzed for parameters with short holding times (e.g., TSS). A more detailed discussion of possible approaches to obtaining the required data, including the rationale for the selected approach, is presented in Section 5.1.

3.3 Sample Locations

Flow-averaged sample composites will be collected at four stations in the Upper Hudson River. The locations include Fenimore Bridge at Bakers Falls, Rogers Island at Fort Edward, the Thompson Island Dam and the Route 4 Bridge at Waterford as shown in Figure 3-3. Each of these four stations coincide with locations that are currently being sampled as part of the water-transect sampling in the Phase 2A field effort. (A total of 14 locations are being sampled as part of the water-transect sampling, including seven locations on the main axis of the upper Hudson River from Glens Falls to Waterford, one on the Mohawk River, Hoosic River, Batten Kill, and Champlain Canal above Lock 7, and three in the Lower Hudson River. The water-column transect stations in the Upper Hudson River are shown on Figure 3-1. See the Final Phase 2 Work Plan, September 1992.) Recent data indicate that PCBs are not detected at RM 197.0 (Hudson Falls) (GE, 1993). Therefore, the originally proposed Glens Falls station for flow-averaged sampling, which is upstream of Hudson Falls, has been deleted and replaced with the downstream location at Waterford.

3.4 Sample Analyses and Schedule

Flow-averaged samples will be collected for six 15-day periods at each of the four locations. Each sample analysis will include the determination of the following:

- Dissolved-phase PCBs on a congener-specific basis;
- Suspended-matter PCBs on a congener-specific basis;

- Total suspended solids/weight-loss-on-ignition; and
- Dissolved organic carbon.

This sampling plan will generate 24 flow-averaged sample analyses for each of the parameters listed above over the sampling period (4 stations x 6 fifteen day events), excluding duplicates and quality control samples. TSS analyses will also be conducted in discrete grab samples collected at each location on each day of sampling, so 192 discrete samples (8 samples/event x 4 locations x 6 15-day events) will also be generated. In addition, temperature, pH, conductivity and dissolved oxygen will be measured in the field at each location at the time of sample collection.

Sampling procedures are contained in Section 6 of this report. Details of the analyses can be found in the appendices of this volume of the Phase 2B SAP/QAPjP. Two consecutive 15-day sampling periods, constituting essentially one month, are tentatively scheduled for three months between March and July of 1993, meaning that this program will overlap with Phase 2A water-column transect sampling.

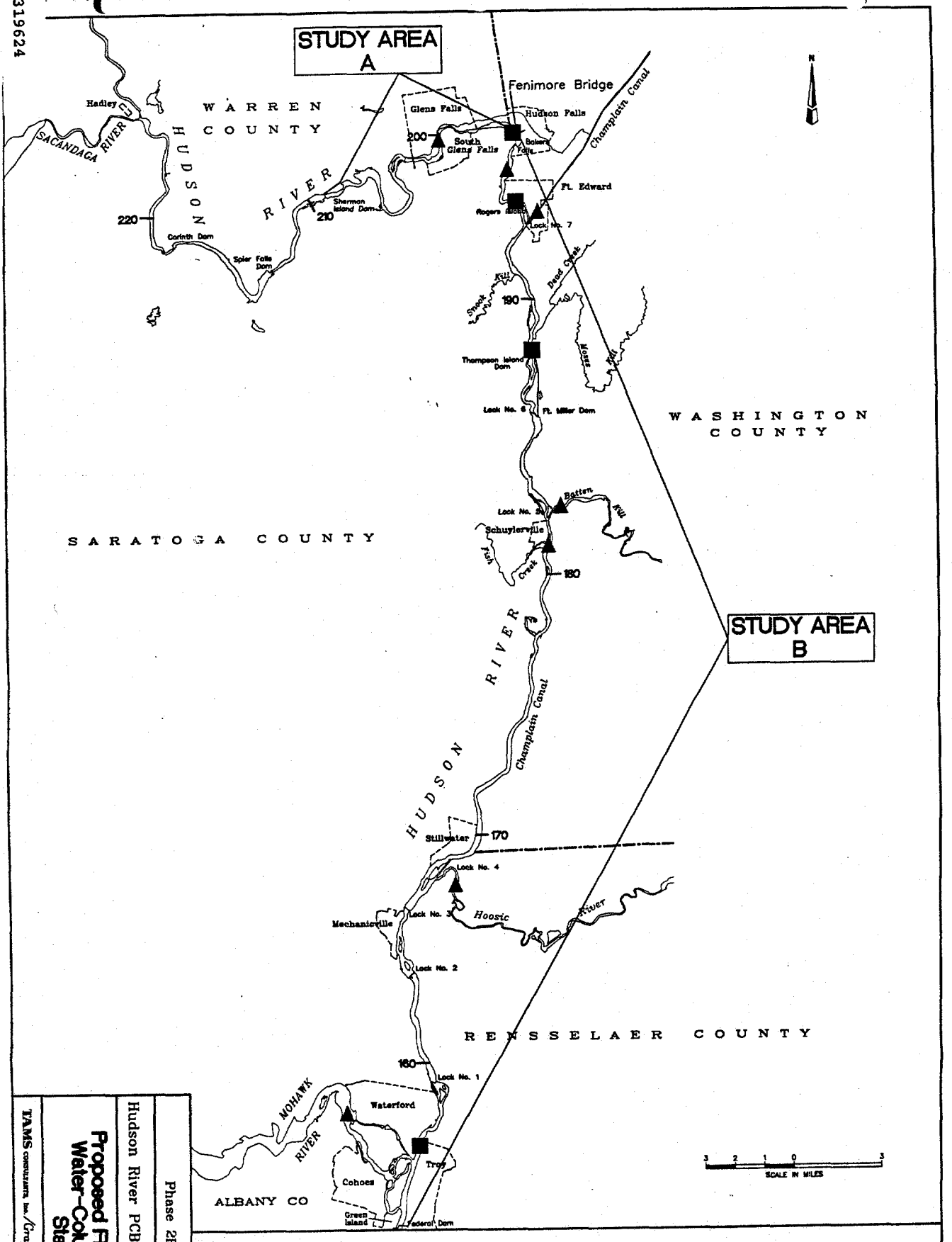
3.4.1 Congener-Specific Water-Column PCB Analyses

Two PCB samples will be derived from each water sample taken for the flow-averaged water-column study: a dissolved-phase PCB sample and a suspended-matter (particulate) phase PCB sample. The congener-specific analysis on the dissolved and suspended-matter fractions will address the following issues:

- the nature of the PCB source(s) to the river, by generating a "fingerprint" based on the congener mixture (e.g., a source derived from an Aroclor-like mixture *vs.* a highly dechlorinated sediment source);

- the effect of *in situ* processes such as gas exchange, aerobic degradation, and particle adsorption on the nature of the PCBs being transported at any given time or location;

- the importance of the lighter congeners in the total PCB mixture borne by the river (previous data suggest that as much as half of the total water column burden may be mono- and dichlorobiphenyls.



Legend

- 180 — River Mile
- ■ — Proposed Combined Water-Column Transect and Flow-Averaged Water-Column Sampling Stations
- ▲ — Proposed Water-Column Transect Sampling Station

Note:

1. Study Area A: Sherman Island Dam to Fenimore Bridge
2. Study Area B: Fenimore Bridge to Federal Dam
3. Actual demarcation between Study Areas A and B is just north of Fenimore Bridge so that Study Area B includes the old Hudson Falls Treatment Plant outfall.
4. USGS flow-monitoring stations are located at Fort Edward near Rogers Island and above Lock 1 near Waterford.

Proposed Flow-Averaged Water-Column Sampling Stations

Hudson River PCB Reassessment RI/FS

Phase 2B SAP/QAPjP

TAMS consultants, Inc./Gradient corporation

Figure 3-1

4 Project Organization

The project team required to perform the flow-averaged water sampling investigation will consist of representatives from USEPA Region II, TAMS Consultants, Inc. and Gradient Corporation, technical consultants, subcontractors performing the field sampling, and analytical laboratories. A Phase 2B flow-averaged sampling project organization chart is provided in Figure 4-1.

The TAMS Project Manager, Albert DiBernardo, reports directly to Douglas Tomchuk, the USEPA Remedial Project Manager (RPM). TAMS will provide overall project management services for the Phase 2B sampling activities. Gradient Corporation, subcontractors to TAMS, will provide technical consulting services for chemistry and laboratory activities. Field samples for the flow-averaged water sampling will be collected and filtered by staff of Rensselaer Polytechnic Institute's (RPI) Department of Earth and Environmental Science under the supervision of TAMS personnel.

4.1 Operations Responsibility

The TAMS Project Manager (PM) is responsible for overseeing the activities of the field team, headed by the Field Operations Leader (FOL), Richard Bopp, who is responsible for proper completion of the tasks included in the Phase 2B SAP/QAPjP. The Field Operations Leader is responsible for making field decisions regarding all field activities. Together with the Field Sampling Coordinator, they (the PM and FOL) are responsible for the field team maintaining proper sampling and decontamination procedures in collecting water and particulate samples, and for following the field measurement protocols outlined in this SAP/QAPjP. Once samples have been collected, the Field Operations Leader will verify that samples are properly packaged and shipped to the analytical laboratories.

4.2 Laboratory Responsibilities

The TAMS/Gradient Quality Assurance Officer (QAO), Dr. A. Dallas Wait, will monitor the activities of the TAMS-contracted analytical laboratories. He is responsible for overseeing the implementation of the technical protocols and documentation requirements of sample analyses in accordance with this SAP/QAPjP.

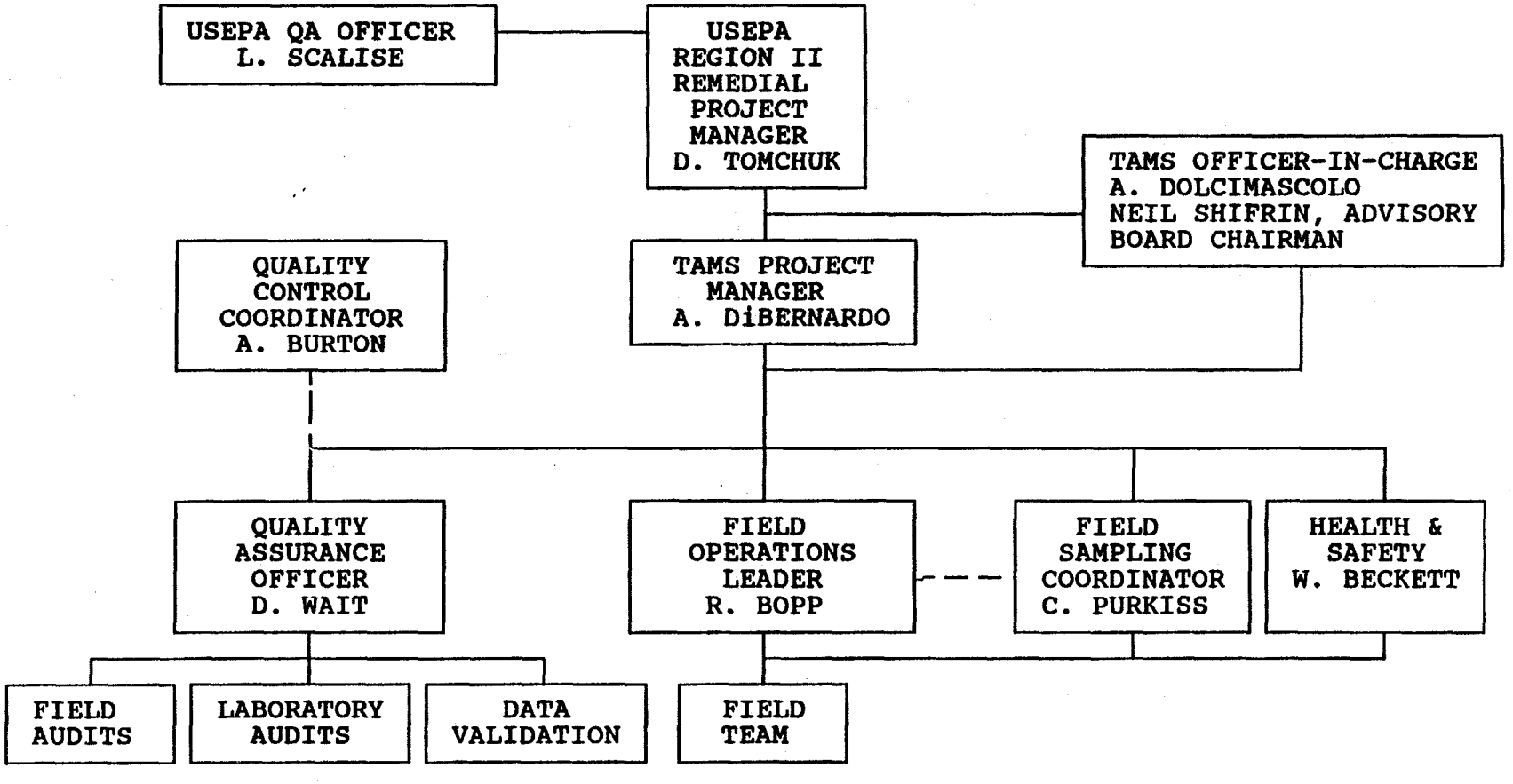
The TAMS/Gradient Quality Assurance Officer will be involved with the selection of TAMS-contracted laboratories. Selection criteria may include a pre-award audit of the laboratory. Criteria to be used in the audit evaluation will be similar to that used by EPA to audit CLP laboratories.

4.3 Quality Assurance Responsibilities

The TAMS/Gradient Field Operations Leader and Field Sampling Coordinator are responsible for maintaining chain-of-custody on all samples collected, as well as verification with sampling team personnel that sampling techniques and quality control procedures are in order before initiation of site activities. They are responsible for prompt review of any quality control deviations at the site. The TAMS/Gradient Quality Assurance Officer will oversee quality control/quality assurance issues for the field operation and the contract laboratories. In addition, each laboratory chosen to perform the analysis will have its own QA Director to monitor internal quality control. The EPA Region II Quality Assurance Officer for this project, Laura Scalise, will be involved with the approval of this SAP/QAPjP, and then monitor its implementation.

Validation of PCB congener data will be the responsibility of USEPA. Validation will be in accordance with the protocols developed by TAMS/Gradient and approved by USEPA specifically for this project (Appendices A-6 and A-7). Validation of other data (dissolved organic carbon, total suspended solids, and weight loss on ignition) will be the responsibility of TAMS/Gradient personnel under the direction of the project QAO or TAMS' ARCS Quality Control Coordinator, Allen Burton.

FIGURE 4-1
HUDSON RIVER REASSESSMENT RI/FS
PHASE 2B FLOW-AVERAGED SAMPLING PROJECT ORGANIZATION



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5 Quality Assurance Objectives for Measurement Data

The primary objective of the Quality Assurance (QA) program is to provide data of sufficient quality and quantity to achieve the objectives as stated in Section 3. A further discussion of the data quality necessary to achieve these objectives is presented below (Section 5.1). Data quality and quantity are measured through comparison of resulting data with established acceptable limits for data precision, accuracy, representativeness, comparability, and completeness (PARCC) as described in "Data Quality Objectives for Remedial Response Activities" (USEPA, 1987a). Analytical sensitivity, evidenced by the method detection limit, is also an important consideration for this project, especially for congener-specific PCB analysis. Data that have certain aspects that may be outside the QA objectives will be evaluated to determine the extent to which the data can be defensibly used to meet the RI/FS objectives. Objectives for the PARCC and sensitivity parameters for this RI/FS are described in this section.

5.1 Data Quality Objectives

As discussed in Section 3.2 of this SAP/QAPjP, the primary objective of the flow-averaged sampling is to obtain data on the total mass flow of PCBs down the Hudson River at several locations. Determination of this requires two pieces of information: the total concentration of PCB congeners in the water column (both dissolved and suspended); and the associated flow rate of the water.

The flow rate of the water for the three northernmost locations will be determined from USGS gaging station data from the station just north of Rogers Island. Flow data for the Waterford sampling location will be obtained from the USGS Waterford staff gage, or from the manual USGS gage at Schuylerville or Fort Edward, if scaling data for Waterford is unavailable (see Section 6).

Aqueous PCB congener concentration data will be obtained by collection and analysis of samples from four locations.

Three approaches to the collection of representative samples for the determination of PCB mass flow were considered. Probably the most accurate method of obtaining total PCB flux data would be to collect and analyze samples every day for a full year. In conjunction with the USGS flow information, PCB flux for any

given time period could be estimated. The effect of seasonal and water flow rate variations on the PCB concentrations and flux could be estimated. The disadvantages of this approach are (1) that it is time-consuming, requiring a full year to complete, and (2) it is very resource-intensive, requiring personnel to conduct field sampling for 365 days, and generating over 1400 samples (365 samples, excluding QC samples, at each of four locations) for analysis. Due to schedule constraints and available resources, this approach was not practical. Therefore, several other approaches were considered.

The second approach is to reduce the length of time over which the daily sampling program was conducted to, for example, three months. The three months would not necessarily have to be consecutive. Based on the overall project schedule, field sampling for the flow-averaged sampling must be completed by July 1993; this approach could achieve this. A range of flows could be sampled (e.g., high flow in April, low flow in July, more typical or midrange flow in June, based on the Ft. Edward USGS data [Figure 6-2]), although seasonal variability would not be accounted for. Personnel requirements would be reduced to about 90 days for the field sampling team; about 360 discrete samples (30 samples/month x 3 months x 4 stations) would be generated for analysis. Resource allocations for related work (e.g., data validation) would also be reduced proportionally.

The third approach, and the approach selected for the flow-averaged sampling event, involves generating flow averaged composite samples for PCB congener analysis. The compositing period chosen was 15 days (rather than 30 days) so that maximum holding times of any portion of the composite would not exceed 21 days (15 days in the field, one day for shipping to the laboratory, and 5 days from VTSR at the lab for extraction); therefore, the data should be considered usable based upon holding time criteria defined in the USEPA Region II data validation guidelines (USEPA, 1992a) and not subject to possible rejection due to "gross exceedance" (more than 14 days) of the 7-day from collection to extraction time specified in the Region II guidelines. In order to obtain an accurate measure of the PCB level in a water sample, it is necessary to separate the sample into suspended matter (collected on a filter) and dissolved (filtered) fractions. This is a result of the time required to extract the PCBs borne by the suspended matter. As was determined during the MDL studies done for sediments and suspended matter analyses in the Phase 2A program, greater than 8 hours of soxhlet extraction is required to completely recover PCBs from these matrices. The soxhlet extraction is a relatively rigorous procedure involving extensive suspended matter/solvent contact at elevated temperatures. The MDL studies indicate that this technique achieves a minimum of 77% recovery for the monochlorobiphenyls and typically greater than 90% recovery for the other congeners. Extraction procedures for whole (unseparated) water samples typically involve

a liquid-liquid extraction of an hour or less which is far less rigorous in extracting the suspended matter. Since existing information suggests that 20% to 90% of the PCB load is bound to suspended matter (Bopp et al., 1985), the less rigorous extraction ordinarily performed on water samples is a significant potential source of error. Therefore, the composite samples will be filtered and separate extractions and analyses will be conducted on each phase.

5.2 PARCC and Sensitivity Objectives

PARCC and sensitivity objectives have been developed for waters and particulates based on sample objectives, analytical methods, historical data (examined in a qualitative sense) and published guidelines for the EPA Contract Laboratory Program (CLP) as listed in Section 17 (References). Specific numerical criteria for PARCC and sensitivity goals are developed to achieve the project-specific DQOs.

PARCC objectives for Phase 2B sampling are summarized on Tables 5-1 and 5-2 for the laboratory and field analyses respectively. Sensitivity (detection limit) objectives are listed in Tables 5-3 and 5-4. PARCC comparability and sensitivity objectives should be achieved through the use of standardized sample collection and analysis procedures. The definition and objectives for PARCC and sensitivity parameters are discussed in greater detail below.

5.2.1 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 the average value. Precision is usually expressed in terms of relative percent difference or relative standard deviation. Measurement of precision is dependent upon sampling technique and analytical method. Both sampling and analysis will be as consistent as possible.

Quality control (QC) samples, including field and laboratory (matrix) duplicate samples, and matrix spike and matrix spike duplicate samples will be analyzed and used to measure precision. An additional measure of precision is the comparison of surrogate recoveries between the unspiked, matrix spike, and matrix spike duplicate sample aliquots. A one-in-twenty frequency per matrix will receive a laboratory (matrix) duplicate

analysis (TSS, WLOI and DOC) and matrix spike/matrix spike duplicate (PCB congener analysis).

As described in Section 6 of this SAP/QAPjP, one composited water (approximately 16 liters) and suspended-matter sample will be produced for each of the four locations twice per month (two 15-day composites or covering a total of 30 days). A composited field duplicate (replicate) sample will also be collected at a frequency sufficient to meet the one field duplicate per 20 samples criterion. Field duplicates will also be collected of this frequency for discrete (grab) sample analyses. Field duplicate results will be evaluated during data validation.

It should be noted that the precision objectives shown on Table 5-1 are guidelines. In accordance with USEPA Region II data validation protocols, data may be fully usable even if these objectives are not met.

5.2.2 Accuracy

Accuracy is a measure of the bias in a measurement system which may result from sampling or analytical error. Sources of error that may contribute to poor accuracy are: laboratory error, sampling inconsistency, field contamination, laboratory contamination, handling, matrix interference, and preservation. Field blanks, surrogate spikes, performance evaluation (PE) samples, as well as matrix spike QC samples, will be used to measure accuracy for project samples. In general, field blanks will be collected at a frequency of one per decontamination event for each type of sampling equipment. Field QC sample collection and analysis requirements are discussed in greater detail in Section 11.

It is acknowledged that there may be some loss of accuracy due to the extended field handling time (up to 15 days) for some of the portions which will make up the composite samples for PCB congener and TSS analysis. To compensate for the long PCB handling time consideration was given to preservation of the discrete grab samples with nitric acid (Hermans et al, 1992). However, after discussion with EPA, it was decided not to preserve the samples for the following reasons:

- There is concern that the applicability of the referenced article is limited due to differences between the sample matrix and sample handling in the literature study (Hermans et al, 1992) and the proposed flow-averaged sampling program.

- The evidence for preservation by acidification was based on a single article; no corroborating articles were found. In fact, another article suggested the opposite (Maguire and Tkacz, 1989).

- The proposed program will result in sample holding times (even for the portion collected on the first day of a 15-day period) which will not "grossly exceed" (i.e., exceed by more than 14 days) the extraction or analysis holding time specified in the USEPA Region II organic data validation guidelines (USEPA, 1992a).

- PCBs are relatively resistant to alteration, and no significant changes to the total PCB content of samples are expected. Keeping the samples chilled at 4°C is expected to minimize biological activity and biodegradation of PCBs. Although changes in the equilibrium partitioning of PCBs between the dissolved and suspended phase may occur, this will not affect the total (dissolved plus suspended) PCB mass in the sample. The samples composited for TSS analysis will also exceed the allowable holding time (7 days) for this analysis. However, the primary objective of this analysis is to obtain a TSS concentration to use in calculating the mass of PCBs associated with transport of suspended matter in the Hudson River. Since the PCB concentration in the suspended matter will be determined by a composite which is filtered at least 15 days after the first portion is collected, the accuracy of the calculating the total mass of PCBs associated with suspended matter is improved by performing the suspended solids analysis on a sample subjected to similar handling (e.g., holding time) and compositing.

5.2.3 Representativeness

Representativeness expresses the degree to which sample data represents the characteristics of the medium or matrix from which it is collected. Samples that are considered representative are ones that are properly collected to characterize the nature and extent of contamination at a given location. Therefore, use of composite samples, a high rate of sample replication, and consistent sampling methods will be implemented. Representativeness will be measured by using the methods (*e.g.* sampling, handling, and preserving) specified in this SAP/QAPjP (Section 6). To the extent feasible within the constraints of site access and sampling personnel safety, the samples will be collected from the river in high flow rate areas (i.e., as near the center of the channel as possible). Comparison of the analytical results from field duplicate samples will provide a direct

measure of individual sample representativeness. Field duplicates will be collected at a minimum of one for every 20 samples for both aqueous and suspended matter matrices.

5.2.4 Comparability

Comparability is a qualitative parameter expressing the confidence with which data sets can be compared. Comparability relies upon precision and accuracy to be within appropriate QC limits before the data can be used for comparison of data sets. This will be accomplished through the consistent use of the analytical and sampling methods described in this SAP/QAPjP (Section 6). This includes quantitating PCB congeners through separate dissolved phase and suspended matter analyses, as has been done for previous (Phase 2A) analyses for this project. Additionally, quantitative and qualitative information on comparability will be obtained for the PCB congener analyses in 10% of the particulate (suspended matter) samples and 5% of the waters by GC/ITD confirmation (Appendix A-5).

For this project, internal comparability (both within and between different parts of the program) is of high importance. Specifically, the ability to draw conclusions regarding "dissolved" and "suspended" matter or fractions requires that such data be reported on a consistent basis. Therefore, all sample filtration will be through a 0.7 μm glass fiber filter. It should be noted that TSS results reported on this basis may vary slightly from those generated from the 1.0 to 1.5 μm filters recommended in EPA method 160.2.

5.2.5 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. Deficiencies in the data may be due to sampling techniques, poor accuracy or precision, or laboratory error. While these deficiencies may affect certain aspects of the data, usable data may still be obtained from applicable samples. Completeness is of the utmost concern for Phase 2B samples. The splitting of each monthly composite into two 15-day composites increases the chances of obtaining at least some usable data from each month, even if part of the data is unusable. The goal for completeness, expressed as usable data obtained based on total data generated, is 90%.

5.2.6 Sensitivity

Quantitation limit goals for laboratory and field analyses planned for the Phase 2B flow-averaged effort are shown on Tables 5-3 and 5-4 of this SAP/QAPjP. Quantitation limits may be 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 in the laboratory, only pesticide grade or better solvents will be used, and method blanks must be demonstrated to be free of contamination prior to analysis. Sample/extract cleanups will be performed (specified in the Appendices) to achieve the specified PCB congener detection limits. If the quantitation limits are still not achievable, the usability of the data, with respect to meeting the Phase 2B objectives, will be evaluated.

5.3 Procedures for Monitoring PARCC Parameters

PARCC parameters will be monitored through the use of procedures which have been referred to in Section 5.1. These procedures will include the use of field blanks, laboratory method blanks, field and laboratory duplicates or replicates, matrix spikes, duplicate matrix spikes, surrogate spikes, performance evaluation samples, laboratory control samples, and a careful examination of calibration and check standards. Laboratory Control Samples (LCSs) and performance evaluation (PE) samples are samples containing a known or true value which the laboratory prepares and analyzes concurrently with project samples. LCSs and PE samples are useful in judging analytical accuracy.

5.4 Field Measurements

Measurement data will be generated in many field activities that are incidental to collecting samples for off-site analytical testing or in activities unrelated to sampling. These activities include, but are not limited to, the following:

- Documenting time and weather conditions;
- Locating and determining the depth of sampling stations; and

- Determining temperature, pH, specific conductance, and dissolved oxygen of water samples.

The general QA objective for field measurement data is to obtain reproducible and comparable measurements to a degree of accuracy consistent with the intended use of the data through the documented use of standardized procedures. The procedures for performing these activities and the standardized formats for documenting them are presented in Section 6 of this SAP/QAPjP.

Table 5-1

Accuracy and Precision Objectives for Laboratory Analyses

Parameter	Matrix	Field Duplicate Precision (% RPD)	Sample/MD or MS/MSD[1] Precision (% RPD)	LCS[2] Accuracy (% Recovery)	MS/MSD Accuracy (% Recovery)	Surrogate Accuracy (% Recovery)
PCB Congeners	Water	50	40	60-150	60-150	60-150[3]
	Particulate	50	40	60-150	60-150	60-150[3]
Dissolved Organic Carbon-Persulfate Oxidation	Water	25	25	90-110	NA	NA
Total Suspended Solids	Water	25	20	90-110	NA	NA
Weight-Loss-on-Ignition	Particulate	25[4]	20[4]	90-110	NA	NA

[1] Five percent of PCB congener analyses will be confirmed by GC/ITD (Appendix A-5 of the SAP/QAPjP) with criteria of <50% RPD between methods.
[2] LCS = ICV for conventional parameters; LCS = MSB (matrix spike blank) for PCB congener analysis.
[3] Surrogates are tetrachlorometaxylene (TCMX) and octachloronaphthalene.
[4] Objective is ± 0.5 mg where WLOI is ≤ 2.0 mg.
NA = Not Applicable

Table 5-2

Accuracy and Precision Objectives for Field Analyses

Measurement	Instrument	Precision	Accuracy
pH	Corning Model 103 or Hanna HI 9025	±0.1 pH units	0.1 pH units
Conductivity	YSI Model 33	±10/25/250 µmho/cm[1]	±5/25/250 µumho/cm[1]
Temperature	YSI Model 33	±0.1°C	±0.1°C or 1% (whichever is greater)
Dissolved Oxygen	YSI Model 57 or YSI Model 51B	0.1 mg/L	±0.1 mg/L at full scale

[1]Depends on scale being used

Table 5-3

PCB Congeners - Detection Limit Goals

Matrix	Homolog	Detection Limit Goal
Particulates	Monochlorobiphenyl	2 ng/filter
	Dichlorobiphenyl through Hexachlorobiphenyl	1 ng/filter
	Heptachlorobiphenyl through Decachlorobiphenyl	1-2 ng/filter
Water (16 liters)	Monochlorobiphenyl	0.1 ng/L
	Dichlorobiphenyl through Hexachlorobiphenyl	0.05 ng/L
	Heptachlorobiphenyl through Decachlorobiphenyl	0.05-0.1 ng/L

Table 5-4

Detection Limit Goals for Conventional Parameters

Parameter	Detection Limit Goal
Dissolved Organic Carbon-Persulfate Oxidation	0.25 mg/L
Total Suspended Solids	0.5 mg/L (on a 1.0 liter sample)
Weight-Loss-on-Ignition	0.5 mg (1% WLOI on a 50 mg TSS sample)

6 Sampling Procedures

The water-column samples will be collected and composited so as to generate a flow-averaged sample. A scale of sample volume to river discharge has been developed (see Section 6.2), which will be used to determine the volume of sample to be collected at each station on any sampling day. Prior to the collection of a day's samples, the USGS monitoring stations in the study area (Fort Edward and Waterford) will be queried to obtain the most recent flow conditions. With these instantaneous flow readings, an appropriate volume of sample will be obtained from each of the four stations. At the end of each 15 day period (8 sampling events), the individual samples will be combined and filtered, producing a single flow-averaged sample (both a dissolved phase and suspended-matter phase) for each station. In this manner, the sample will have sufficient volume to permit the measurement of PCB congeners at the required detection limits.

This technique avoids the inherent day-to-day variability in water-column levels, which has been noted in the historical data, by creating a flow-averaged sample for each location. As discussed in Section 5.1, it also avoids the large analytical costs involved in establishing a sufficiently large database of daily samples to permit a statistically valid analysis of the mean PCB loads.

6.1 Sample Locations

The four flow-averaged water sample locations are shown and described in Section 3.3 of this report. The sample locations have been selected based upon information collected in the Interim Phase 1 Report (TAMS, 1992) and other recent data (GE, 1993).

6.2 Sample Collection

Flow-averaged composite samples will be generated for PCB congeners, dissolved organic carbon, total suspended solids (TSS) and weight loss on ignition (WLOI). In addition, discrete grab samples will also be analyzed for TSS in order to meet holding time criteria. A summary of the planned samples is presented as Table 6-1.

It is anticipated that the composited samples for PCB analysis will be approximately 15 to 20 liters in volume (as is the case for the water-transect sample). Thus, a two liter sample collected every other day will yield 16 liters over the 15-day sampling period. A sample consisting of exactly two liters will be collected on a day where the river flow matches the average monthly flow rate based on historic USGS records at the Fort Edward monitoring station. A sample larger than two liters will be collected for flows above the average and samples less than two liters will be collected for daily flows below the average. A scaling system as described below will be used to determine the volume to be collected.

Historical data at the USGS Fort Edward gaging station will be used to determine the appropriate scaling for the three northernmost stations (see Figure 6-1 and Table 6-2a). This gaging station is used since it is located within the area of study. There are no significant flow inputs between this monitoring station and the sample collection locations (Bakers Falls, Fort Edward and the Thompson Island Dam). The Fort Edward USGS gaging station will be queried electronically, prior to each day's sampling, to obtain the most recent flow conditions to determine the sample volume to be collected that day from each station.

For scaling the Waterford sampling location, it is anticipated that either the USGS staff gage at Waterford or the nearby NYSDOT staff gage, calibrated against historical USGS flow gaging data, will be used (Table 6-2b). (The Waterford USGS gaging station is currently operative but may be altered due to planned construction at the site.) If data specific to the Waterford Station are not available, then the next available upstream USGS station (Stillwater, Schuylerville or Fort Edward) will be used to estimate the Waterford flow.

It is anticipated that the flow-averaged water sampling program will take place between March and July, 1993. The scaling for these months based on monthly averages of flow is shown on Table 6-2a for the USGS Fort Edward Station and on Table 6-2b for the USGS Waterford Station.

6.2.1 PCB Sample Collection

The procedure for collecting the approximate 16 liter composite sample, to be analyzed for dissolved phase and particulate (suspended matter) PCBs, is outlined below:

1. The final sampling access points will be defined prior to the flow-averaged sampling during the Phase 2A water-transect sampling.

2. Each day, prior to sampling, determine the appropriate sample volume to be collected as described above using Tables 6-2 and 6-3.

3. Collect each sample for PCB analysis directly into clean, prepared 1-liter, 500 mL, or 120 mL bottles. Bottle requirements are dependent on river flow, and are shown on Table 6-3. The sample bottle cleaning procedure is described in Section 6.4.2 of this SAP/QAPjP.

4. Sample bottles will be filled by one of two means, based on site accessibility.

   -- From bridges (Bakers Falls [Fennimore Bridge] and Waterford sample locations), lower the sample bottle(s) to the appropriate depth, when possible halfway between the water surface and the river bottom, but at least 6 inches below the water surface and approximately in the center of the channel. Remove the stopper or invert the bottle and allow the bottle to fill. When these methods can not be implemented, the bottles may be lowered to the water surface in an upright position then plunged rapidly to the appropriate sampling depth and allowed to fill.

   -- When sampling from shore (Fort Edward and Thompson Island), the samples bottles will be submerged using a pole or other means, so as to obtain a sample as far from shore as possible.

5. Immediately after sample collection, the samples for PCB analysis will be cooled to 4°C.

6. Cap, label, custody seal, and place each bottle on ice until all bottles have been filled to collect the required volume for the day based on the scale above.

7. Measure conductivity, temperature, pH and DO at each sampling station during the sample collection period, and record the measurements in the field notebook.

8. Repeat these procedures at the remaining three stations.

9. The daily samples for PCB analysis will be refrigerated and stored at Rensselaer Polytechnic Institute for the remainder of the 15-day sampling period before they are filtered. The samples will be filtered within 24 hours of the last day of each 15-day sampling event at the interim laboratory (RPI) prior to shipment to the analytical laboratory.

6.2.2 PCB Sample Filtration

On the 16th day of the sampling period (after 8 sampling events), each 16 liter sample (consisting of various 1-liter, 500 mL, and 120 mL bottles) must be separated into dissolved and suspended matter fractions. Approximately five empty, pre-cleaned 4-liter amber glass bottles are necessary for performing the filtration step. (The number of 4-liter bottles required is dependent upon the total flow-averaged volume collected over the 15-day sampling period. A total of five 4-liter bottles will be needed if the actual volume collected is close to the 16-liter nominal sample volume.) The filtration procedure, based on the assumption of a 16-liter flow-averaged sample, is described below.

1. Assemble a 6 in. stainless steel filter housing and rinse the housing with Hudson River water to equilibrate the housing with PCBs in the water. Place a clean, pre-fired, pre-weighed 6 in. Whatman glass fiber filter, grade GF/F (0.7 μm) or equivalent, in the filter housing. The filter is pre-fired in clean, PCB free air at 450 °C overnight.

2. Pass approximately 3 1/2 to 4-liters of water from the first set of sample bottles through the filter under pressure using a pump, or by pressurizing the holding container with air. If air is used to displace the liquid, then a magnetic stirring rod will be used to keep the suspended matter in suspension.

3. Collect the first 3 1/2 to 4 liters of filtrate in an empty, labelled 4-liter bottle. Rinse the first set of bottles, now empty (constituting the 4 liters), with a small amount of filtrate (100-200 mL) to recover any additional suspended matter remaining in the bottle. This step is repeated as necessary until no suspended material is visible on the surface of the bottle. The filtrate

used to rinse the sample bottles will be added to the filter and passed through the filter with the next set of sample bottles (see step 4). Save the empty bottles for later hexane rinsing (step 8). The first 4-liter bottle is used to hold the initial filtrate which will be used for subsequent sample bottle rinsing.

4. Before beginning to filter the remaining sample bottles, charge the empty 4-liter receiving bottles with approximately 15 mL of pesticide-grade hexane. Collect the next 3 to 3 1/2 liters of filtrate into the second bottle. Rinse the original sample bottles with filtrate from the first 4-liter bottle until no suspended matter remains. Re-filter the filtrate used to rinse the original bottles. Save the empty bottles for subsequent rinsing with hexane (step 8).

5. Repeat step 4 until all 16 liters of the sample have been filtered. This should yield a total of four charged 4-liter bottles of filtrate and one uncharged 4-liter bottle of filtrate for a 16 liter sample.

6. Charge the first 4-liter bottle (i.e., the initial filtrate) with approximately 15 mL of hexane. All five 4-liter bottles now have a 15 mL hexane addition.

7. It may be necessary to use a second filter if the first filter becomes clogged. Both filters will then be treated as one suspended matter sample.

8. Using a minimum amount of solvent, rinse the original (and now empty) sample bottles (from steps 3 and 4) with three separate hexane rinses, using 20 mL for each rinse. During a given rinse step the hexane aliquot can be used to rinse all bottles at that step (i.e., a single 20 mL aliquot can be used to rinse all empty bottles for rinse number one). After each rinse collect the hexane and add it to one of the 4-liter sample bottles. (Distribute the heane rinses as evenly as practical among the five 4-liter bottles so that each bottle has approximately the same amount of hexane.) This step is intended to remove any PCBs which may adhere to the walls of the original bottles.

9. Seal the five 4-liter bottles containing the sample filtrate and hexane rinses for shipment to the subcontract laboratory for PCB analysis.

10. Place the filters containing the suspended matter in a labelled, clean glass jar for shipment to the subcontract laboratory for PCB congener analysis of the particulate fraction. A separate glass jar will be used for each particulate filter sample.

6.2.3 Flow-Averaged Sample Collection for Total Suspended Solids and Weight Loss on Ignition Analyses

Additional aliquots of water will be collected each day at each of the four sites for compositing into flow-averaged samples for analysis of total suspended solids/weight-loss-on-ignition and dissolved organic carbon.

The total suspended solids (TSS) and weight-loss-on-ignition (WLOI) analyses will be performed on the suspended-matter fraction of a water sample, to be collected as follows:

1. Collect each sample for TSS/WLOI analysis at each of the four sampling stations on each sampling day (i.e., every other day) using a clean 500 mL (or 1 liter) container by submersion to the same approximate depth as the PCB sample. Unlike the PCB samples, the volume collected will be independent of instantaneous flow (i.e., no scale).

2. Cap, custody seal, and place each bottle on ice. Samples will be refrigerated at RPI prior to filtration. TSS and WLOI samples will not be preserved.

3. Repeat steps 1 and 2 above for the next 7 sampling days. This will yield a total of eight 500 mL (or one liter) samples per location for the sampling period.

4. Upon completion of the 8 sampling events, the samples will be composited based on flow into a single flow-weighted mixture by stirring each jar and removing the required sample volume based on flow from each jar using a graduated cylinder (see Tables 6-3a to 6-3g). The measured individual sample volumes are then composited.

5. This flow-weighted mixture is then filtered through a glass-fiber filter using the same procedure and apparatus as for the PCB samples, discussed above. This suspended matter sample will then be analyzed for TSS and WLOI, as detailed in the Appendices.

6. Thus, two composited samples will be collected at each of the four stations per month (two 15-day periods), for a total of eight flow-averaged TSS/WLOI samples per month, excluding QA samples.

6.2.4 Collection of Flow-Averaged Samples for Dissolved Organic Carbon Analysis and Discrete Grab Samples for Total Suspended Solids (TSS) and Weight-Loss-on-Ignition (WLOI) Analysis

The flow-averaged water samples for dissolved organic carbon (DOC) and discrete grab samples for total suspended solids (TSS) and weight-loss-on-ignition (WLOI) will be collected from the water samples as follows:

1. Collect each sample for flow-averaged DOC analysis and discrete TSS sample analysis at each of the four sampling stations on each sampling day (i.e., every other day) using a clean 1-liter bottle. Samples are collected by submersion to the same approximate depth as the PCB sample. Unlike the PCB samples, the volume collected will be independent of instantaneous flow (i.e., no scale).

2. Cap, custody seal, and place each bottle on ice. Samples will be refrigerated at RPI prior to filtration.

3. Filter the entire 1-liter samples within 24 hours of collection through a clean, pre-weighed, Whatman GF/F 0.7 μm glass fiber filter as described above (Section 6.2.3). Collect the filtered water in a clean bottle and preserve by acidification to a pH $\leq$2 with sulfuric acid. The final pH will be confirmed with pH paper.

4. Cap, seal, refrigerate and store each of the four bottles with the filtered water at RPI.

5. The filters may be stored in glass jars and sealed for later TSS analysis. Dry and weigh the filters (as per Method 160.2) to determine the TSS within the specified holding time.

6. WLOI analysis will be performed only on samples from which at least 50 mg TSS is obtained.

7. Repeat steps 1 through 6 above for the next 7 sampling days. This will yield a total of eight approximate 1-liter filtered samples per location, and eight discrete grab samples per location analyzed for TSS. Depending on the TSS in the river from zero to eight discrete grab samples from each 15-day sampling event will be analyzed for WLOI.

8. At this point after the first eight sampling events, the preserved DOC filtrates will be composited based on flow into a single flow-weighted mixture by using a graduated cylinder to collect the required sample volume based on flow (see Table 6-3) from each bottle and composite. The extra sample volume may be discarded.

9. Thus, two flow-averaged samples for DOC analysis will be collected at each of the four stations per month, for a total of eight DOC samples per month, excluding QC samples. The samples will be analyzed utilizing the DOC persulfate oxidation method as detailed in Appendix B-1. 64 discrete grab samples per month (8 samples/15-day event x 2 15-day events/month x 4 locations) will be generated for TSS analysis.

6.3 Sample Containers, Preservation, and Holding Times

Due to the nature of the flow-averaged sampling technique, some portion of the composited samples will be held beyond the standard USEPA holding time of seven days from collection to extraction for aqueous PCB analysis of unpreserved samples. The samples will be extracted such that no sample portion exceeds the USEPA 21 day holding time criteria beyond which the subsequent analysis may be rejected. Standard USEPA data validation criteria state that PCB results from samples held between 7 and 21 days may be considered estimated depending upon the professional judgement of the data validator (USEPA, 1992a). The laboratory will be required to begin the PCB sample extraction within 5 days of verified time of sample receipt (VTSR). This requirement serves to yield samples which will have undergone laboratory extraction within the time interval

of 21 days or less between collection of the first sample in a composite and its extraction. The results of the flow-averaged samples for PCB analysis will meet the data quality objectives by meeting the following criteria:

- Samples collected every other day for 15 days (8 sampling events) are cooled to 4°C upon collection.
- Samples are composited, filtered and subsequently charged with hexane on the sixteenth day of sample collection.
- Samples are extracted within six days of shipping from the interim laboratory (i.e., 5 days from VTSR at the subcontract laboratory facility).

Data quality for samples which do not meet these criteria will be evaluated by the data validation personnel and qualified based on the professional judgment of the validator.

In a similar manner, the holding times for total suspended solids (by USEPA method 160.2) will be exceeded for the composited samples. To address this concern, discrete grab samples for TSS will be collected and analyzed for each day of operation within the standard (7-day) holding time to provide accurate values of the total suspended solids levels. However, in order to properly represent the composited suspended matter PCB sample, a composited total suspended solids sample will be collected and handled in a similar manner, i.e., this sample will be produced at the end of each 15-day sampling period (on the sixteenth day) along with the composited suspended matter PCB sample. This analysis will permit a more accurate calculation of the suspended matter-borne PCB content of the sample.

DOC samples have a standard holding time of 28 days (USEPA, 1983). Daily samples collected to produce a composite will be filtered within 24 hours and acidified as noted in Section 6.2.4. The compositing will be completed within 24 hours of collection of the final sample in a 15-day event. However, in order that all portions of the composited DOC sample meet the holding time, the composited sample must be analyzed within 12 days of compositing.

Weight loss on ignition analysis has no specified holding time.

The specific containers, preservatives, and holding times that will be utilized for this investigation are presented in Table 6-4.

6.4 Preparation of Sampling Equipment and Containers

6.4.1 Decontamination of Sampling Equipment

Decontamination and subsequent use of decontaminated equipment will be documented in a field notebook. If visual signs such as discolorations indicate that decontamination was insufficient, the equipment will be decontaminated again. If the situation persists, the equipment will be taken out of service. Decontaminated stainless steel equipment will be wrapped in aluminum foil when not in use.

Field decontamination of stainless steel apparatus used to collect samples for PCB analysis will consist of rinsing with potable tap water, followed by acetone rinse and distilled deionized analyte free water rinse. When potable tap water is unavailable, Hudson River water will be used instead. After decontamination, stainless steel apparatus and utensils will be allowed to dry and then be wrapped and stored in aluminum foil.

Filtering apparatus will be decontaminated in the laboratory prior to each the completion of the 16 day sampling period. A sufficient number of filtering apparatus (approximately 6 units) will be prepared to complete a full set of samples, duplicates and blanks. The filtering apparatus will be decontaminated using the procedure outlined in Section 6.4.2.

6.4.2 Preparation of Sample Containers and Filtration Apparatus

Filtration apparatus and glass containers supplied for PCB congener sampling and transport of aqueous samples will be precleaned using the following procedure:

1. Wash with tap water and laboratory soap, followed by extensive tap water rinse.

2. Rinse with distilled water (three times).

3. Rinse twice with acetone (pesticide grade).

4. Rinse twice with hexane (pesticide grade).

5. Rinse twice with acetone (pesticide grade).

6. Stand inverted for 20 minutes to permit acetone to drain.

7. Heat in large, low temperature (60° C) oven at least six hours to remove last traces of organic solvents, or (for stainless steel equipment only) allow to air dry in a controlled environment for at least 12 hours.

8. Cool and cover with aluminum foil previously rinsed with hexane.

9. Secure aluminum foil cover with rubber band.

Glassware for other analyses (TSS, DOC, WLOI) will be purchased pre-cleaned according to EPA requirements (e.g., Eagle-Picher Level 1 or equivalent) and used with no further clean-up. Sample bottles will be obtained from a commercial source and will meet the cleaning and documentation requirements of the "Statement of Work for Maintenance of a Quality-Controlled Prepared Sample Container Repository" (USEPA, 1987b, as revised).

6.5 Collection of Field Blanks

Field blanks will be collected, filtered, handled, and preserved in the same manner as the corresponding environmental samples. Field blank sample volume will be the same as for the corresponding sample. Field blanks will be collected and analyzed for PCB congeners (filtered water and particulates); dissolved organic carbon in water; and total suspended solids. The TSS field blank will be analyzed for WLOI only if more than 0.5 mg TSS are detected.

A PCB congener field blank will consist of 16-liters of laboratory-provided analyte free water which is passed through the filtration apparatus (Section 6.2.2) and collected in four 4-liter bottles to which approximately 30 mL of pesticide grade hexane has been added. The glass fiber filter is extracted and analyzed as the particulate PCB congener field blank.

Field blanks for other parameters will be collected in a similar manner, except the analyte free water will be obtained from a commercial source. The DOC field blank will be preserved to pH 2 with H_2SO_4. Field blanks for other parameters will not be preserved.

6.6 Sample Handling and Shipment

All flow-averaged water samples collected in the field will be placed in the appropriate containers and shipped directly to the Interim Laboratory (RPI) for storage and refrigeration as well as subsequent filtration when appropriate. The DOC samples will remain at RPI for analysis. Filtered particulate samples will be shipped directly to the subcontract laboratory for PCB congener analysis of the aqueous and particulate fractions. The TSS samples (both the discrete grab samples and the flow-averaged samples) and the WLOI samples will remain at RPI for analysis.

All sample containers will be properly labelled prior to shipment. At a minimum, the sample label will contain:

- The Investigation Name (Hudson River Phase 2B)
- Field Sample Number
- Sample Tag Number (PCB analysis only)
- Date and Time Collected
- Matrix
- Sampler's Name
- Preservatives Added (if applicable)
- Analysis Parameters
- Remarks

Detailed protocols for sample packaging and shipping are found in Appendix C-1.

6.7 Sample Custody

Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a completed, signed chain of custody form enclosed in each sample cooler. A copy of the chain of custody form will be retained by the Field Team Leader. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. Chain of custody procedures are detailed in Section 7 of this SAP/QAPjP.

Table 6-1

Approximate Number of Samples to be Collected during Phase 2B Flow-Averaged Sampling *

A. FLOW-AVERAGED SAMPLES

Analytical Procedure	Flow-Averaged Water Sampling	
	Field Samples/Event (SDG)	Total field Samples (6 events)
PCB, water (filtered/dissolved)	4	24
PCB, non-filterable (particulate filter)	4	24
Dissolved Organic Carbon (DOC) by Persulfate Oxidation Method	4	24
Total Suspended Solids (TSS)/Weight-Loss-On-Ignition (WLOI)	4	24

B. DISCRETE GRAB SAMPLES

Analytical Procedure**	Flow-Averaged Water Sampling	
	Field Samples/Event (SDG)	Total field Samples (6 events)
Total Suspended Solids	32	192
Weight Loss on Ignition	TBD	TBD

* Note: Excluding QA/QC Samples (blanks and MS/MSD or MD). A full set of QC samples (1 field blank (as applicable); 1 matrix spike; and 1 matrix spike duplicate (MSD applicable to PCB analysis only) will be collected for each SDG (15-day event); field duplicates will be submitted at a rate of one per 20 samples (a total of 2 field duplicates for each flow-averaged sampling analytical parameter; an estimated total of 10 field duplicates discrete grab samples for TSS).

** No discrete grab samples will be analyzed for PCBs or DOC.

TBD To be determined. WLOI analysis will be performed only on discrete grab samples in which at least 50 mg of suspended solids are obtained.

A Sample Delivery Group (SDG) is defined here as one set of 4 field samples, collected from a single 15-day sampling event.

Table 6-2a

Scaling System for Bakers Falls, Fort Edward, and Thompson Island Dam Sampling Stations

Based on USGS Fort Edward Data (1977-1990)

MONTH	MEAN FLOW (cfs)	MEDIAN FLOW (cfs)	SCALED FLOW (CFS) = TWO LITER PCB SAMPLE 160 mL DOC/TSS SAMPLE	TABLE REFERENCE FOR DAILY FLOW-AVERAGED SAMPLE VOLUME TO BE COLLECTED
March	6,162	5,180	5,000	6-3C
April	10,105	9,100	10,000	6-3F
May	7,645	5,600	6,000	6-3D
June	4,179	3,430	4,000	6-3B
July	2,808	2,730	3,000	6-3A

Table 6-2b

Scaling System for Waterford Sampling Station

Based on USGS Waterford Data (1977-1990)

MONTH	MEAN FLOW (cfs)	MEDIAN FLOW (cfs)	SCALED FLOW (CFS) = TWO LITER PCB SAMPLE 160 mL DOC/TSS SAMPLE	TABLE REFERENCE FOR DAILY FLOW-AVERAGED SAMPLE VOLUME TO BE COLLECTED
March	11,112	8,040	8,000	6-3E
April	15,083	13,400	14,000	6-3G
May	10,119	7,210	8,000	6-3E
June	6,153	4,970	5,000	6-3C
July	3,763	3,370	4,000	6-3B

TABLE 6-3A
PHASE 2B FLOW-AVERAGED SAMPLING VOLUME DETERMINATION

SCALED MEAN FLOW = 3,000 cfs

MEAN PCB VOLUME/DAY = 2,000 mL
MEAN DOC VOLUME/DAY = 160 mL
MEAN TSS VOLUME/DAY = 480 mL

FLOW INCREMENT = 188 cfs
PCB INCREMENT = 125 mL
DOC INCREMENT = 10 mL
TSS INCREMENT = 30 mL

ACTUAL FLOW RANGE (cfs)	PCB VOLUME (mL)	NUMBER OF PCB BOTTLES 1 LITER	500 mL	125 mL	DOC VOLUME * (mL)	TSS VOLUME ** (mL)
0 - 94	0	0	0	0	0	0
94 - 281	125	0	0	1	10	30
281 - 469	250	0	0	2	20	60
469 - 656	375	0	0	3	30	90
656 - 844	500	0	1	0	40	120
844 - 1,031	625	0	1	1	50	150
1,031 - 1,219	750	0	1	2	60	180
1,219 - 1,406	875	0	1	3	70	210
1,406 - 1,594	1,000	1	0	0	80	240
1,594 - 1,781	1,125	1	0	1	90	270
1,781 - 1,969	1,250	1	0	2	100	300
1,969 - 2,156	1,375	1	0	3	110	330
2,156 - 2,344	1,500	1	1	0	120	360
2,344 - 2,531	1,625	1	1	1	130	390
2,531 - 2,719	1,750	1	1	2	140	420
2,719 - 2,906	1,875	1	1	3	150	450
2,906 - 3,094	2,000	2	0	0	160	480
3,094 - 3,281	2,125	2	0	1	170	510
3,281 - 3,469	2,250	2	0	2	180	540
3,469 - 3,656	2,375	2	0	3	190	570
3,656 - 3,844	2,500	2	1	0	200	600
3,844 - 4,031	2,625	2	1	1	210	630
4,031 - 4,219	2,750	2	1	2	220	660
4,219 - 4,406	2,875	2	1	3	230	690
4,406 - 4,594	3,000	3	0	0	240	720
4,594 - 4,781	3,125	3	0	1	250	750
4,781 - 4,969	3,250	3	0	2	260	780
4,969 - 5,156	3,375	3	0	3	270	810
5,156 - 5,344	3,500	3	1	0	280	840
5,344 - 5,531	3,625	3	1	1	290	870
5,531 - 5,719	3,750	3	1	2	300	900
5,719 - 5,906	3,875	3	1	3	310	930
5,906 - 6,094	4,000	4	0	0	320	960
6,094 - 6,281	4,125	4	0	1	330	990
6,281 - 6,469	4,250	4	0	2	340	1020
6,469 - 6,656	4,375	4	0	3	350	1050
6,656 - 6,844	4,500	4	1	0	360	1080
6,844 - 7,031	4,625	4	1	1	370	1110
7,031 - 7,219	4,750	4	1	2	380	1140
7,219 - 7,406	4,875	4	1	3	390	1170
7,406 - 7,594	5,000	5	0	0	400	1200
7,594 - 7,781	5,125	5	0	1	410	1230
7,781 - 7,969	5,250	5	0	2	420	1260
7,969 - 8,156	5,375	5	0	3	430	1290
8,156 - 8,344	5,500	5	1	0	440	1320
8,344 - 8,531	5,625	5	1	1	450	1350
8,531 - 8,719	5,750	5	1	2	460	1380
8,719 - 8,906	5,875	5	1	3	470	1410
8,906 - 9,094	6,000	6	0	0	480	1440

NOTES:

* One liter water samples will be collected at each station each sampling day. These samples will be filtered to generate the daily TSS sample and a portion of the composite DOC sample. The DOC samples will be composited from the daily portions using a graduated cylinder based based on the tabulated DOC volumes listed.

** One (or two) liter water samples will be collected at each station each sampling day. These samples will be combined to yield a composite TSS/WLOI sample for each station using a graduated cylinder based on the tabulated TSS volumes listed.

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TABLE 6-3B
PHASE 2B FLOW-AVERAGED SAMPLING VOLUME DETERMINATION

SCALED MEAN FLOW = 4,000 cfs

MEAN PCB VOLUME/DAY = 2,000 mL
MEAN DOC VOLUME/DAY = 160 mL
MEAN TSS VOLUME/DAY = 480 mL

FLOW INCREMENT = 250 cfs
PCB INCREMENT = 125 mL
DOC INCREMENT = 10 mL
TSS INCREMENT = 30 mL

ACTUAL FLOW RANGE (cfs)	PCB VOLUME (mL)	NUMBER OF PCB BOTTLES 1 LITER	500 mL	125 mL	DOC VOLUME * (mL)	TSS VOLUME ** (mL)
0 - 125	0	0	0	0	0	0
125 - 375	125	0	0	1	10	30
375 - 625	250	0	0	2	20	60
625 - 875	375	0	0	3	30	90
875 - 1,125	500	0	1	0	40	120
1,125 - 1,375	625	0	1	1	50	150
1,375 - 1,625	750	0	1	2	60	180
1,625 - 1,875	875	0	1	3	70	210
1,875 - 2,125	1,000	1	0	0	80	240
2,125 - 2,375	1,125	1	0	1	90	270
2,375 - 2,625	1,250	1	0	2	100	300
2,625 - 2,875	1,375	1	0	3	110	330
2,875 - 3,125	1,500	1	1	0	120	360
3,125 - 3,375	1,625	1	1	1	130	390
3,375 - 3,625	1,750	1	1	2	140	420
3,625 - 3,875	1,875	1	1	3	150	450
3,875 - 4,125	2,000	2	0	0	160	480
4,125 - 4,375	2,125	2	0	1	170	510
4,375 - 4,625	2,250	2	0	2	180	540
4,625 - 4,875	2,375	2	0	3	190	570
4,875 - 5,125	2,500	2	1	0	200	600
5,125 - 5,375	2,625	2	1	1	210	630
5,375 - 5,625	2,750	2	1	2	220	660
5,625 - 5,875	2,875	2	1	3	230	690
5,875 - 6,125	3,000	3	0	0	240	720
6,125 - 6,375	3,125	3	0	1	250	750
6,375 - 6,625	3,250	3	0	2	260	780
6,625 - 6,875	3,375	3	0	3	270	810
6,875 - 7,125	3,500	3	1	0	280	840
7,125 - 7,375	3,625	3	1	1	290	870
7,375 - 7,625	3,750	3	1	2	300	900
7,625 - 7,875	3,875	3	1	3	310	930
7,875 - 8,125	4,000	4	0	0	320	960
8,125 - 8,375	4,125	4	0	1	330	990
8,375 - 8,625	4,250	4	0	2	340	1020
8,625 - 8,875	4,375	4	0	3	350	1050
8,875 - 9,125	4,500	4	1	0	360	1080
9,125 - 9,375	4,625	4	1	1	370	1110
9,375 - 9,625	4,750	4	1	2	380	1140
9,625 - 9,875	4,875	4	1	3	390	1170
9,875 - 10,125	5,000	5	0	0	400	1200
10,125 - 10,375	5,125	5	0	1	410	1230
10,375 - 10,625	5,250	5	0	2	420	1260
10,625 - 10,875	5,375	5	0	3	430	1290
10,875 - 11,125	5,500	5	1	0	440	1320
11,125 - 11,375	5,625	5	1	1	450	1350
11,375 - 11,625	5,750	5	1	2	460	1380
11,625 - 11,875	5,875	5	1	3	470	1410
11,875 - 12,125	6,000	6	0	0	480	1440

NOTES:

* One liter water samples will be collected at each station each sampling day. These samples will be filtered to generate the daily TSS sample and a portion of the composite DOC sample. The DOC samples will be composited from the daily portions using a graduated cylinder based based on the tabulated DOC volumes listed.

** One (or two) liter water samples will be collected at each station each sampling day. These samples will be combined to yield a composite TSS/WLOI sample for each station using a graduated cylinder based on the tabulated TSS volumes listed.

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TABLE 6-3C
PHASE 2B FLOW-AVERAGED SAMPLING VOLUME DETERMINATION
SCALED MEAN FLOW = 5,000 cfs

MEAN PCB VOLUME/DAY = 2,000 mL
MEAN DOC VOLUME/DAY = 160 mL
MEAN TSS VOLUME/DAY = 480 mL

FLOW INCREMENT = 313 cfs
PCB INCREMENT = 125 mL
DOC INCREMENT = 10 mL
TSS INCREMENT = 30 mL

ACTUAL FLOW RANGE (cfs)	PCB VOLUME (mL)	NUMBER OF PCB BOTTLES 1 LITER	500 mL	125 mL	DOC VOLUME * (mL)	TSS VOLUME ** (mL)
0 - 156	0	0	0	0	0	0
156 - 469	125	0	0	1	10	30
469 - 781	250	0	0	2	20	60
781 - 1,094	375	0	0	3	30	90
1,094 - 1,406	500	0	1	0	40	120
1,406 - 1,719	625	0	1	1	50	150
1,719 - 2,031	750	0	1	2	60	180
2,031 - 2,344	875	0	1	3	70	210
2,344 - 2,656	1,000	1	0	0	80	240
2,656 - 2,969	1,125	1	0	1	90	270
2,969 - 3,281	1,250	1	0	2	100	300
3,281 - 3,594	1,375	1	0	3	110	330
3,594 - 3,906	1,500	1	1	0	120	360
3,906 - 4,219	1,625	1	1	1	130	390
4,219 - 4,531	1,750	1	1	2	140	420
4,531 - 4,844	1,875	1	1	3	150	450
4,844 - 5,156	2,000	2	0	0	160	480
5,156 - 5,469	2,125	2	0	1	170	510
5,469 - 5,781	2,250	2	0	2	180	540
5,781 - 6,094	2,375	2	0	3	190	570
6,094 - 6,406	2,500	2	1	0	200	600
6,406 - 6,719	2,625	2	1	1	210	630
6,719 - 7,031	2,750	2	1	2	220	660
7,031 - 7,344	2,875	2	1	3	230	690
7,344 - 7,656	3,000	3	0	0	240	720
7,656 - 7,969	3,125	3	0	1	250	750
7,969 - 8,281	3,250	3	0	2	260	780
8,281 - 8,594	3,375	3	0	3	270	810
8,594 - 8,906	3,500	3	1	0	280	840
8,906 - 9,219	3,625	3	1	1	290	870
9,219 - 9,531	3,750	3	1	2	300	900
9,531 - 9,844	3,875	3	1	3	310	930
9,844 - 10,156	4,000	4	0	0	320	960
10,156 - 10,469	4,125	4	0	1	330	990
10,469 - 10,781	4,250	4	0	2	340	1020
10,781 - 11,094	4,375	4	0	3	350	1050
11,094 - 11,406	4,500	4	1	0	360	1080
11,406 - 11,719	4,625	4	1	1	370	1110
11,719 - 12,031	4,750	4	1	2	380	1140
12,031 - 12,344	4,875	4	1	3	390	1170
12,344 - 12,656	5,000	5	0	0	400	1200
12,656 - 12,969	5,125	5	0	1	410	1230
12,969 - 13,281	5,250	5	0	2	420	1260
13,281 - 13,594	5,375	5	0	3	430	1290
13,594 - 13,906	5,500	5	1	0	440	1320
13,906 - 14,219	5,625	5	1	1	450	1350
14,219 - 14,531	5,750	5	1	2	460	1380
14,531 - 14,844	5,875	5	1	3	470	1410
14,844 - 15,156	6,000	6	0	0	480	1440

NOTES:

* One liter water samples will be collected at each station each sampling day. These samples will be filtered to generate the daily TSS sample and a portion of the composite DOC sample. The DOC samples will be composited from the daily portions using a graduated cylinder based based on the tabulated DOC volumes listed.

** One (or two) liter water samples will be collected at each station each sampling day. These samples will be combined to yield a composite TSS/WLOI sample for each station using a graduated cylinder based on the tabulated TSS volumes listed.

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TABLE 6-3D
PHASE 2B FLOW-AVERAGED SAMPLING VOLUME DETERMINATION

SCALED MEAN FLOW = 6,000 cfs

		FLOW INCREMENT =	375 cfs
MEAN PCB VOLUME/DAY =	2,000 mL	PCB INCREMENT =	125 mL
MEAN DOC VOLUME/DAY =	160 mL	DOC INCREMENT =	10 mL
MEAN TSS VOLUME/DAY =	480 mL	TSS INCREMENT =	30 mL

ACTUAL FLOW RANGE (cfs)	PCB VOLUME (mL)	NUMBER OF PCB BOTTLES 1 LITER	500 mL	125 mL	DOC VOLUME * (mL)	TSS VOLUME ** (mL)
0 - 188	0	0	0	0	0	0
188 - 563	125	0	0	1	10	30
563 - 938	250	0	0	2	20	60
938 - 1,313	375	0	0	3	30	90
1,313 - 1,688	500	0	1	0	40	120
1,688 - 2,063	625	0	1	1	50	150
2,063 - 2,438	750	0	1	2	60	180
2,438 - 2,813	875	0	1	3	70	210
2,813 - 3,188	1,000	1	0	0	80	240
3,188 - 3,563	1,125	1	0	1	90	270
3,563 - 3,938	1,250	1	0	2	100	300
3,938 - 4,313	1,375	1	0	3	110	330
4,313 - 4,688	1,500	1	1	0	120	360
4,688 - 5,063	1,625	1	1	1	130	390
5,063 - 5,438	1,750	1	1	2	140	420
5,438 - 5,813	1,875	1	1	3	150	450
5,813 - 6,188	2,000	2	0	0	160	480
6,188 - 6,563	2,125	2	0	1	170	510
6,563 - 6,938	2,250	2	0	2	180	540
6,938 - 7,313	2,375	2	0	3	190	570
7,313 - 7,688	2,500	2	1	0	200	600
7,688 - 8,063	2,625	2	1	1	210	630
8,063 - 8,438	2,750	2	1	2	220	660
8,438 - 8,813	2,875	2	1	3	230	690
8,813 - 9,188	3,000	3	0	0	240	720
9,188 - 9,563	3,125	3	0	1	250	750
9,563 - 9,938	3,250	3	0	2	260	780
9,938 - 10,313	3,375	3	0	3	270	810
10,313 - 10,688	3,500	3	1	0	280	840
10,688 - 11,063	3,625	3	1	1	290	870
11,063 - 11,438	3,750	3	1	2	300	900
11,438 - 11,813	3,875	3	1	3	310	930
11,813 - 12,188	4,000	4	0	0	320	960
12,188 - 12,563	4,125	4	0	1	330	990
12,563 - 12,938	4,250	4	0	2	340	1020
12,938 - 13,313	4,375	4	0	3	350	1050
13,313 - 13,688	4,500	4	1	0	360	1080
13,688 - 14,063	4,625	4	1	1	370	1110
14,063 - 14,438	4,750	4	1	2	380	1140
14,438 - 14,813	4,875	4	1	3	390	1170
14,813 - 15,188	5,000	5	0	0	400	1200
15,188 - 15,563	5,125	5	0	1	410	1230
15,563 - 15,938	5,250	5	0	2	420	1260
15,938 - 16,313	5,375	5	0	3	430	1290
16,313 - 16,688	5,500	5	1	0	440	1320
16,688 - 17,063	5,625	5	1	1	450	1350
17,063 - 17,438	5,750	5	1	2	460	1380
17,438 - 17,813	5,875	5	1	3	470	1410
17,813 - 18,188	6,000	6	0	0	480	1440

NOTES:

* One liter water samples will be collected at each station each sampling day. These samples will be filtered to generate the daily TSS sample and a portion of the composite DOC sample. The DOC samples will be composited from the daily portions using a graduated cylinder based based on the tabulated DOC volumes listed.

** One (or two) liter water samples will be collected at each station each sampling day. These samples will be combined to yield a composite TSS/WLOI sample for each station using a graduated cylinder based on the tabulated TSS volumes listed.

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TABLE 6-3E
PHASE 2B FLOW-AVERAGED SAMPLING VOLUME DETERMINATION
SCALED MEAN FLOW = 8,000 cfs

MEAN PCB VOLUME/DAY = 2,000 mL
MEAN DOC VOLUME/DAY = 160 mL
MEAN TSS VOLUME/DAY = 480 mL

FLOW INCREMENT = 500 cfs
PCB INCREMENT = 125 mL
DOC INCREMENT = 10 mL
TSS INCREMENT = 30 mL

ACTUAL FLOW RANGE (cfs)	PCB VOLUME (mL)	NUMBER OF PCB BOTTLES 1 LITER	500 mL	125 mL	DOC VOLUME * (mL)	TSS VOLUME ** (mL)
0 - 250	0	0	0	0	0	0
250 - 750	125	0	0	1	10	30
750 - 1,250	250	0	0	2	20	60
1,250 - 1,750	375	0	0	3	30	90
1,750 - 2,250	500	0	1	0	40	120
2,250 - 2,750	625	0	1	1	50	150
2,750 - 3,250	750	0	1	2	60	180
3,250 - 3,750	875	0	1	3	70	210
3,750 - 4,250	1,000	1	0	0	80	240
4,250 - 4,750	1,125	1	0	1	90	270
4,750 - 5,250	1,250	1	0	2	100	300
5,250 - 5,750	1,375	1	0	3	110	330
5,750 - 6,250	1,500	1	1	0	120	360
6,250 - 6,750	1,625	1	1	1	130	390
6,750 - 7,250	1,750	1	1	2	140	420
7,250 - 7,750	1,875	1	1	3	150	450
7,750 - 8,250	2,000	2	0	0	160	480
8,250 - 8,750	2,125	2	0	1	170	510
8,750 - 9,250	2,250	2	0	2	180	540
9,250 - 9,750	2,375	2	0	3	190	570
9,750 - 10,250	2,500	2	1	0	200	600
10,250 - 10,750	2,625	2	1	1	210	630
10,750 - 11,250	2,750	2	1	2	220	660
11,250 - 11,750	2,875	2	1	3	230	690
11,750 - 12,250	3,000	3	0	0	240	720
12,250 - 12,750	3,125	3	0	1	250	750
12,750 - 13,250	3,250	3	0	2	260	780
13,250 - 13,750	3,375	3	0	3	270	810
13,750 - 14,250	3,500	3	1	0	280	840
14,250 - 14,750	3,625	3	1	1	290	870
14,750 - 15,250	3,750	3	1	2	300	900
15,250 - 15,750	3,875	3	1	3	310	930
15,750 - 16,250	4,000	4	0	0	320	960
16,250 - 16,750	4,125	4	0	1	330	990
16,750 - 17,250	4,250	4	0	2	340	1020
17,250 - 17,750	4,375	4	0	3	350	1050
17,750 - 18,250	4,500	4	1	0	360	1080
18,250 - 18,750	4,625	4	1	1	370	1110
18,750 - 19,250	4,750	4	1	2	380	1140
19,250 - 19,750	4,875	4	1	3	390	1170
19,750 - 20,250	5,000	5	0	0	400	1200
20,250 - 20,750	5,125	5	0	1	410	1230
20,750 - 21,250	5,250	5	0	2	420	1260
21,250 - 21,750	5,375	5	0	3	430	1290
21,750 - 22,250	5,500	5	1	0	440	1320
22,250 - 22,750	5,625	5	1	1	450	1350
22,750 - 23,250	5,750	5	1	2	460	1380
23,250 - 23,750	5,875	5	1	3	470	1410
23,750 - 24,250	6,000	6	0	0	480	1440

NOTES:

* One liter water samples will be collected at each station each sampling day. These samples will be filtered to generate the daily TSS sample and a portion of the composite DOC sample. The DOC samples will be composited from the daily portions using a graduated cylinder based based on the tabulated DOC volumes listed.

** One (or two) liter water samples will be collected at each station each sampling day. These samples will be combined to yield a composite TSS/WLOI sample for each station using a graduated cylinder based on the tabulated TSS volumes listed.

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TABLE 6-3F
PHASE 2B FLOW-AVERAGED SAMPLING VOLUME DETERMINATION

SCALED MEAN FLOW = 10,000 cfs

MEAN PCB VOLUME/DAY = 2,000 mL
MEAN DOC VOLUME/DAY = 160 mL
MEAN TSS VOLUME/DAY = 480 mL

FLOW INCREMENT = 625 cfs
PCB INCREMENT = 125 mL
DOC INCREMENT = 10 mL
TSS INCREMENT = 30 mL

ACTUAL FLOW RANGE (cfs)	PCB VOLUME (mL)	NUMBER OF PCB BOTTLES 1 LITER	500 mL	125 mL	DOC VOLUME * (mL)	TSS VOLUME ** (mL)
0 - 313	0	0	0	0	0	0
313 - 938	125	0	0	1	10	30
938 - 1,563	250	0	0	2	20	60
1,563 - 2,188	375	0	0	3	30	90
2,188 - 2,813	500	0	1	0	40	120
2,813 - 3,438	625	0	1	1	50	150
3,438 - 4,063	750	0	1	2	60	180
4,063 - 4,688	875	0	1	3	70	210
4,688 - 5,313	1,000	1	0	0	80	240
5,313 - 5,938	1,125	1	0	1	90	270
5,938 - 6,563	1,250	1	0	2	100	300
6,563 - 7,188	1,375	1	0	3	110	330
7,188 - 7,813	1,500	1	1	0	120	360
7,813 - 8,438	1,625	1	1	1	130	390
8,438 - 9,063	1,750	1	1	2	140	420
9,063 - 9,688	1,875	1	1	3	150	450
9,688 - 10,313	2,000	2	0	0	160	480
10,313 - 10,938	2,125	2	0	1	170	510
10,938 - 11,563	2,250	2	0	2	180	540
11,563 - 12,188	2,375	2	0	3	190	570
12,188 - 12,813	2,500	2	1	0	200	600
12,813 - 13,438	2,625	2	1	1	210	630
13,438 - 14,063	2,750	2	1	2	220	660
14,063 - 14,688	2,875	2	1	3	230	690
14,688 - 15,313	3,000	3	0	0	240	720
15,313 - 15,938	3,125	3	0	1	250	750
15,938 - 16,563	3,250	3	0	2	260	780
16,563 - 17,188	3,375	3	0	3	270	810
17,188 - 17,813	3,500	3	1	0	280	840
17,813 - 18,438	3,625	3	1	1	290	870
18,438 - 19,063	3,750	3	1	2	300	900
19,063 - 19,688	3,875	3	1	3	310	930
19,688 - 20,313	4,000	4	0	0	320	960
20,313 - 20,938	4,125	4	0	1	330	990
20,938 - 21,563	4,250	4	0	2	340	1020
21,563 - 22,188	4,375	4	0	3	350	1050
22,188 - 22,813	4,500	4	1	0	360	1080
22,813 - 23,438	4,625	4	1	1	370	1110
23,438 - 24,063	4,750	4	1	2	380	1140
24,063 - 24,688	4,875	4	1	3	390	1170
24,688 - 25,313	5,000	5	0	0	400	1200
25,313 - 25,938	5,125	5	0	1	410	1230
25,938 - 26,563	5,250	5	0	2	420	1260
26,563 - 27,188	5,375	5	0	3	430	1290
27,188 - 27,813	5,500	5	1	0	440	1320
27,813 - 28,438	5,625	5	1	1	450	1350
28,438 - 29,063	5,750	5	1	2	460	1380
29,063 - 29,688	5,875	5	1	3	470	1410
29,688 - 30,313	6,000	6	0	0	480	1440

NOTES:

* One liter water samples will be collected at each station each sampling day. These samples will be filtered to generate the daily TSS sample and a portion of the composite DOC sample. The DOC samples will be composited from the daily portions using a graduated cylinder based based on the tabulated DOC volumes listed.

** One (or two) liter water samples will be collected at each station each sampling day. These samples will be combined to yield a composite TSS/WLOI sample for each station using a graduated cylinder based on the tabulated TSS volumes listed.

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TABLE 6-3G
PHASE 2B FLOW-AVERAGED SAMPLING VOLUME DETERMINATION
SCALED MEAN FLOW = 14,000 cfs

MEAN PCB VOLUME/DAY = 2,000 mL
MEAN DOC VOLUME/DAY = 160 mL
MEAN TSS VOLUME/DAY = 480 mL

FLOW INCREMENT = 875 cfs
PCB INCREMENT = 125 mL
DOC INCREMENT = 10 mL
TSS INCREMENT = 30 mL

ACTUAL FLOW RANGE (cfs)	PCB VOLUME (mL)	NUMBER OF PCB BOTTLES 1 LITER	500 mL	125 mL	DOC VOLUME * (mL)	TSS VOLUME ** (mL)
0 - 438	0	0	0	0	0	0
438 - 1,313	125	0	0	1	10	30
1,313 - 2,188	250	0	0	2	20	60
2,188 - 3,063	375	0	0	3	30	90
3,063 - 3,938	500	0	1	0	40	120
3,938 - 4,813	625	0	1	1	50	150
4,813 - 5,688	750	0	1	2	60	180
5,688 - 6,563	875	0	1	3	70	210
6,563 - 7,438	1,000	1	0	0	80	240
7,438 - 8,313	1,125	1	0	1	90	270
8,313 - 9,188	1,250	1	0	2	100	300
9,188 - 10,063	1,375	1	0	3	110	330
10,063 - 10,938	1,500	1	1	0	120	360
10,938 - 11,813	1,625	1	1	1	130	390
11,813 - 12,688	1,750	1	1	2	140	420
12,688 - 13,563	1,875	1	1	3	150	450
13,563 - 14,438	2,000	2	0	0	160	480
14,438 - 15,313	2,125	2	0	1	170	510
15,313 - 16,188	2,250	2	0	2	180	540
16,188 - 17,063	2,375	2	0	3	190	570
17,063 - 17,938	2,500	2	1	0	200	600
17,938 - 18,813	2,625	2	1	1	210	630
18,813 - 19,688	2,750	2	1	2	220	660
19,688 - 20,563	2,875	2	1	3	230	690
20,563 - 21,438	3,000	3	0	0	240	720
21,438 - 22,313	3,125	3	0	1	250	750
22,313 - 23,188	3,250	3	0	2	260	780
23,188 - 24,063	3,375	3	0	3	270	810
24,063 - 24,938	3,500	3	1	0	280	840
24,938 - 25,813	3,625	3	1	1	290	870
25,813 - 26,688	3,750	3	1	2	300	900
26,688 - 27,563	3,875	3	1	3	310	930
27,563 - 28,438	4,000	4	0	0	320	960
28,438 - 29,313	4,125	4	0	1	330	990
29,313 - 30,188	4,250	4	0	2	340	1020
30,188 - 31,063	4,375	4	0	3	350	1050
31,063 - 31,938	4,500	4	1	0	360	1080
31,938 - 32,813	4,625	4	1	1	370	1110
32,813 - 33,688	4,750	4	1	2	380	1140
33,688 - 34,563	4,875	4	1	3	390	1170
34,563 - 35,438	5,000	5	0	0	400	1200
35,438 - 36,313	5,125	5	0	1	410	1230
36,313 - 37,188	5,250	5	0	2	420	1260
37,188 - 38,063	5,375	5	0	3	430	1290
38,063 - 38,938	5,500	5	1	0	440	1320
38,938 - 39,813	5,625	5	1	1	450	1350
39,813 - 40,688	5,750	5	1	2	460	1380
40,688 - 41,563	5,875	5	1	3	470	1410
41,563 - 42,438	6,000	6	0	0	480	1440

NOTES:

* One liter water samples will be collected at each station each sampling day. These samples will be filtered to generate the daily TSS sample and a portion of the composite DOC sample. The DOC samples will be composited from the daily portions using a graduated cylinder based based on the tabulated DOC volumes listed.

** One (or two) liter water samples will be collected at each station each sampling day. These samples will be combined to yield a composite TSS/WLOI sample for each station using a graduated cylinder based on the tabulated TSS volumes listed.

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Table 6-4
Sample Containers, Preservation, and Holding Times

Parameter	Matrix	Holding Time	Container	Preservative	Sample Size
PCB Congener	Water	7(21)/40[1] days	4 Liter Amber Glass bottles[2]	Cool to 4°C (hexane after filtration)[5]	16 Liters
PCB Congener	Particulate Filter	7(21)/40[1] days	500 mL Glass jar for filter	temp ≅ 4°C	80-200 mg (estimated)
Dissolved Organic Carbon	Water	28 days[3]	250 mL glass bottle	H_2SO_4 pH ≤2 temp ≅ 4°C	1 Liter
Total Susp. Solids	Water	7 days[4]	1 liter Plastic Bottle	temp ≅ 4°C	1 Liter
Weight Loss On Ignition	Particulate	none	Glass Bottle	temp ≅ 4°C	20-50 mg (estimated)

NOTES

1. Seven days is the sample holding time (from collection) specified in USEPA Region II data validation guidelines. This holding time may be exceeded by up to 14 days before the holding time is considered "grossly exceeded". Therefore, the holding time goal for this study is that no portion of the flow-averaged composite exceeds 21 days (7 days plus 14 days) prior to extraction. Laboratory extractions are to be performed within five days of VTSR at the laboratory. 40 days is the holding time for analysis of the sample extract.

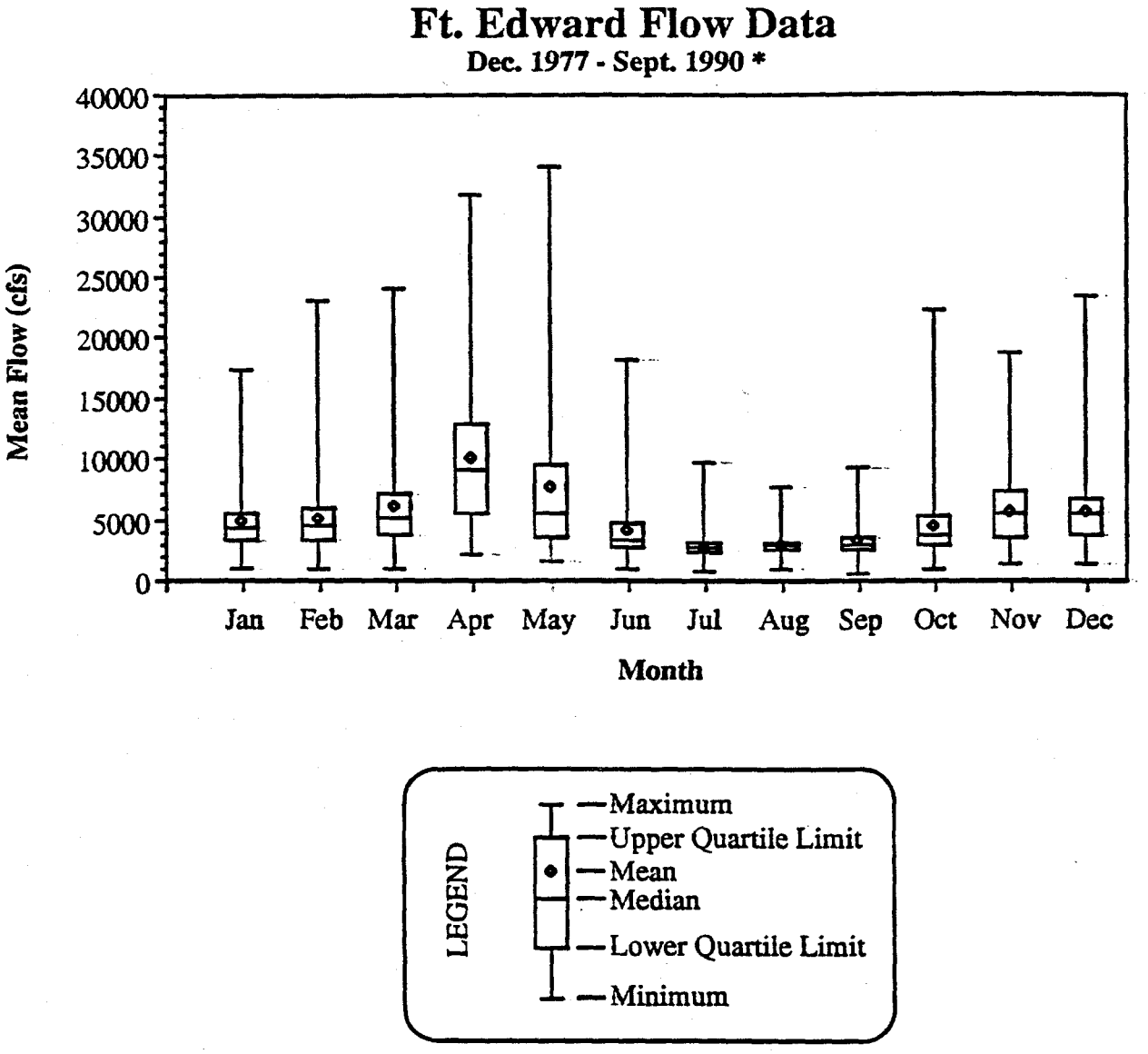
2. Sample is collected into 1-liter, 500 mL, and 120 mL glass bottles, filtered, and then shipped in 4-liter bottles. Based on river flow rate, actual number of bottles may vary.

3. In order to achieve this holding time (28 days from sample collection) for all portions of the composited samples, the laboratory must analyze these samples within 12 days of compositing.

4. Seven days is the holding time for discrete (daily) grab samples. The holding time objective for flow-averaged composite samples is 5 days from VTSR, so TSS data are comparable to PCB congener data.

5. Hexane is not considered a preservative but is used to extract residual solids and PCBs which may adhere to sample bottle walls.

Figure 6-1



* From U.S.G.S. Water Resources Data - New York - Water Years 1977 to 1990 - Vol. 1. - Eastern New York excluding Long Island (1978 - 1991).

7 Chain of Custody Procedures

An essential part of any sampling/analytical scheme is the ability to document the history of samples. This is begun as soon as the samples are in custody. A sample is in custody when it meets any one of the following requirements:

- It is in your actual possession, or
- It is in your view after being in your physical possession, or
- It was in your possession, and then you locked or sealed it to prevent tampering, or
- It is in a secure area.

Chain of custody establishes the documentation and control necessary to identify and trace a sample from collection to final analysis. Such documentation includes labeling to prevent mix-up, container seals to prevent unauthorized tampering with contents, secure custody, and the necessary records to support potential litigation. These precautions are crucial for a valid chain of custody. It is policy to follow the USEPA sample custody or chain of custody protocol as described in "NEIC Policies and Procedures," (EPA, revised May 1986). This custody is in three parts: sample collection; laboratory; custody; and final evidence files. Final evidence files, including all originals of laboratory reports, are maintained under document control in a secure area. The original laboratory reports will be placed in the final evidence files six months after completion of the final report.

The anticipated sample custody is outlined as follows: Daily grab samples for all parameters (DOC, TSS/WLOI, and PCBs) will be collected by RPI staff and transported back to RPI. At RPI, the samples will be logged into the laboratory sample control system, placed in a controlled storage area, and refrigerated as needed. Within the appropriate schedule, samples will be filtered, composited and analyzed at the RPI laboratory. Those samples scheduled to be shipped to the laboratory performing the PCB analyses (i.e., particulate filters and filtered 16-liter water samples) will shipped from RPI and will accompanied by a standard USEPA SAS packing list/chain-of-custody form (Figure 7-1), or equivalent.

7.1 Sample Identification

In order to properly track all samples collected for the flow-averaged sampling investigation, a 10 character alphanumeric identification system will be used. This system is based on earlier sampling efforts (e.g., high resolution coring and water column sampling). The sample numbering system will provide a quick source of information on sample type and is being followed for all sample collection.

The sample numbering format is defined as follows:

AA-000-0000A

where "A" represents a letter and "0" represents a number.

The first two characters represent the sampling effort. For this sampling effort, all samples will be labelled FA for unfiltered aqueous whole water samples; FW for filtered water samples, and FS for suspended matter (filtered particulates). Each of the next three digits has a specific meaning. The first digit identifies the sequential 15-day sampling event (1 through 6). The second digit is a field duplicate identifier: "2" indicates a duplicate, "0" for all other samples. The third digit represents the sequential day (1 through 8) of a 15-day event, or composite (designated by "9"). The final four digits are the station location number. Stations will be numbered in the same manner as for the water column transect sampling. It should be noted that these sample locations are not the same as those used for the ecological sampling program (Volume 2 of the Phase 2B SAP/QAPjP). Therefore, the flow-averaged sample locations will be designated as follows:

Location Name	Station Location Number
Bakers Falls (Fenimore Bridge)	0002
Fort Edward (Rogers Island/Rt 197)	0004
Thompson Island Dam	0005
Waterford	0008

The first three digits will be used to differentiate between samples at each sampling station. For example, the first daily sample will be #01, the second daily sample will be #02, and so on (through #08) where # refers to the 15-day event (i.e., events 1 to 6). Composite samples will be designated as #09. The last alphanumeric character is a letter used to designate composite (C), field duplicates (D), matrix spike samples (M) (this designation also applies to samples designated for matrix duplicate and matrix spike duplicate analysis), or field blanks (B). Field duplicates will have the number "2" as the second digit of the initial 3-digit sequence (e.g., sample FS-321-0002C is a field duplicate of FS-301-0002C).

For example, sample number FW-309-0002C would be the composite filtered water sample taken at the station location #02 (Bakers Falls) for the third 15-day event of the flow-averaged sampling program. The field duplicate of this sample would be designated FW-329-0002C. The discrete grab samples which were composited to produce the composite sample are labelled FA-301-0002, FA-302-0002, FA-303-0002, FA-304-0002, FA-305-0002, FA-306-0002, FA-307-0002 and FA-308-0002. The composite suspended matter sample would be labelled FS-309-0002C. Samples collected in separate containers for individual analyses (e.g., PCB congener, DOC, etc.) will be recorded in the field log book and marked on the chain-of-custody forms.

7.2 Field Specific Custody Procedures

Sampling team personnel will perform the sampling and will retain custody until shipment to the laboratory. One chain-of-custody form will be used for each sample shuttle (cooler) shipped to the laboratory. Figure 7-1 provides a sample of a chain-of-custody form.

The field activities will be recorded daily in a serialized field logbook. The following information will be recorded in the logbook used at this site:

- Where, exactly, was the sample taken?
- Who took the sample, and who witnessed it?
- Date and time of sample collection.
- Sample number, airbill number, seal number.
- Sampling conditions, i.e., type of material, weather on-site, type of sampling container and preparation, description of sampling procedure, preservation, and shipping.

The sample packaging and shipment procedures, detailed in Appendix C-1 and summarized below, will be performed so that the samples will arrive at the laboratory with the chain of custody intact.

Field procedures will be as follows:

- The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.

- All bottles will be tagged with sample numbers and locations (see Section 7.1).

- Sample tags will be completed for each sample using waterproof ink.

- The Field Operations Leader must review field activities to determine whether proper custody procedures were followed during the field work.

Transfer of custody and shipment procedures will be as follows:

- Samples will be accompanied by a properly completed chain of custody form. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving shall sign, date, and note the time on the record. This record documents transfer of custody of samples from sampler to another person, to the interim laboratory (RPI), to the analytical laboratory, or to/from a secure storage area.

- Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with the completed, signed chain of custody form enclosed in each sample box or cooler. Shipping containers will be secured with strapping tape or duct tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and front left of the cooler. The custody seals will be covered with clear plastic tape. The cooler will be strapped shut with strapping or duct tape in at least two locations.

- All shipments will be accompanied by the chain of custody record identifying the contents. The original record will accompany the shipment, and a copy will be retained by the Field Operations Leader.

- If the samples are sent by common carrier, an airbill or bill of lading will be used. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the custody form, as long as the custody forms are sealed inside the sample cooler, and the custody seals remain intact.

7.3 Laboratory Custody Procedures

Samples will be received by the laboratory sample custodian. Samples will be unpacked and inspected for the following:

- Broken or leaking bottles
- Presence of all samples listed on field chain of custody
- Bottle labels match field chain of custody
- Presence of ice and temperature of the cooler
- Number of coolers received matches number shown on airbill

The sample custodian will fill out a Shipment Condition Inspection Report (Figure 7-2) or equivalent. If problems or discrepancies are noted, they will be documented on this form. Discrepancies in the number of samples received or sample bottle labels will also be documented on the field chain of custody form. The sample custodian will then sign and date the field chain of custody form.

After accepting custody of the samples, the sample custodian will log in the samples. Each sample will be assigned a unique sequential laboratory number which will be used for tracking the sample through the laboratory. The field chain of custody, inspection report, and airbill will then be forwarded to the laboratory project manager.

The laboratory project manager or designee will inspect the paperwork. If problems are noted, the laboratory project manager will resolve them with the TAMS project coordinator.

After log-in, samples will be placed in refrigerated storage pending analysis. Sample chain of custody is maintained throughout the laboratory by a system of door locks. Access will require use of a key issued to company employees. Thus, in order to gain access to the laboratories, one must either be an employee or be escorted by an employee.

7.4 Final Evidence File

The final evidence file for the project will consist of: laboratory data packages (summary and raw data from the analysis of QC samples and investigative samples, chromatograms, mass spectra, calibration data, worksheet, sample preparation, chain-of-custody record), logs, field logbooks, pictures and subcontractor reports. All reports will be retained by EPA Region II.

FIGURE 7-1
CHAIN-OF-CUSTODY RECORD

EPA — United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Special Analytical Service
Packing List/Chain of Custody

SAS No.

1. Project Code	Account Code	2. Region No.	Sampling Co.	4. Date Shipped	Carrier
Regional Information		Sampler (Name)		Airbill Number	
Non-Superfund Program		Sampler Signature		5. Ship To	
Site Name		3. Type of Activity (Remedial / Removal)			
City, State	Site Spill ID	SF (Lead) PRP (Pre-Remedial) ST FED	RIFS RD PA RA SSI O&M LSI NPLDL	CLEM REMA REM OIL UST	

6. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil
7. Waste
8. Other (Specify)

7. Preservative (Enter in Column C)
1. HCl
2. HNO_3
3. $NAHSO_4$
4. H_2SO_4
5. NaOH
6. Other (SAS) (Specify)
7. Ice only
N Not preserved

Sample Numbers	A Matrix Enter from Box 6	B Conc Low Med High	C Preservative Used from Box 7	D Analysis	E Sample used for spike and/or duplicate	F Regional Specific Tracking Number or Tag Number	G Station Location Identifier	H Mo/Day/Year/Time Sample Collection	I Sampler Initials	J Designated Field QC
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

Shipment for SAS complete? (Y/N)

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

Split Samples ☐ Accepted (Signature)
☐ Declined

EPA Form
DISTRIBUTION:
White - Region Copy Yellow - SMO Copy Pink - Lab Copy for Return to SMO Gold - Lab Copy

S 004856

Note: This form is used for Special Analytical Services (SAS) laboratories procured through the USEPA Sample Management Office (SMO) Forms used for Routine Analytical Services laboratories procured through USEPA-SMO, and for laboratories contracted directly by TAMS, may vary.

FIGURE 7-2
SAMPLE RECEIVING CHECKLIST

Control #: _____ Date Received: _____
Job Code: _____ Date Inspected: _____
Inspected by: _____ Time Inspected: _____
(print name)

Paperwork	Yes	No	Intact	Broken
Airbill	_____	_____		
Cooler Custody Seals:	_____	_____	_____	_____
Bottle Custody Seals:	_____	_____	_____	_____
Chain-of-Custody:	_____	_____		
Traffic Reports:	_____	_____		
Sample Tags:	_____	_____		
Tags Listed on COC:	_____	_____		

Sample Condition	Cool	Warm	Hot	Degrees C
Cooler Temperature:	_____	_____	_____	_____

	Yes	No	
Ice:	_____	_____	Melted
Bottles Broken:	_____	_____	
Bottles Leaking:	_____	_____	

Preservation pH: (record measured pH)	_____ OK	_____ Not OK	_____ Not Checked
Other _____:	_____	_____	

Shipment Condition:	_____ OK	_____ Not OK	_____ Major	_____ Minor

Problems and Comments

_____
_____
_____
_____

_____ _____
Signature Date

TAMS/*Gradie*

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8 Calibration Procedures and Frequency

8.1 Field Instruments

Field personnel will follow the procedures described in the instrument manufacturer's instructions and the SOPs in Appendices C-2 through C-4 so that measurements during the investigation have been collected with properly calibrated instruments. Field equipment will be calibrated at the frequency shown on Table 8-1, and maintained and repaired in accordance with manufacturer's specifications. In addition, prior to use, each major piece of equipment will be cleaned, decontaminated, checked for damages, and repaired as needed. These activities will be noted in the field log notebook.

Despite even the most rigorous maintenance program, equipment failures do occur. When equipment cannot be repaired in the field, it will be replaced as quickly as possible.

Quality control efforts, accuracy and precision objectives for field measurement equipment are summarized in Section 11. Calibration procedures and frequency for all field instruments are summarized in Table 8-1. Specific detailed methods of calibration for the following instruments are presented in the appendices as follows:

Instrument	Appendix
pH Meter	C-2
Dissolved Oxygen Meter	C-3
Conductivity/Temperature Meter	C-4

8.2 Laboratory Calibration

The analytical methods selected for use in this investigation specify the types and frequency of calibrations. The specific calibration requirements are delineated within the methods provided in the following appendices:

Parameter	Appendix/Reference
PCB Congener (GC/ECD)	A-4
PCB Congeners (GC/ITD confirmation)	A-5
Extraction and Cleanup of Water Samples	A-2
Extraction and Cleanup of Particulate Samples	A-3
Weight-Loss-on-Ignition	B-2
Dissolved Organic Carbon by Persulfate Oxidation	B-1
Total Suspended Solids	MCAWW 160.2, USEPA 1983

Table 8-1

Equipment Maintenance and Calibration Protocols

Equipment	Maintenance/Calibration	Frequency
pH meters (Corning 103 or Hanna HI 9025)	Calibrate with two pH buffer solutions. (pH 7 and 10)	Daily before use. Check at pH 7 every 4 hours.
Temperature	As per manufacturer's instructions	Once per day before use.
Specific conductance (SI - SCT 33)	Calibration verified per manufacturer's instructions. Recalibration, if necessary, will be performed by the manufacturer.	Once per day before use.
Dissolved oxygen meter (YSI 51B or YSI 57)	Calibration according to manufacturer's recommendations with ambient air.	Once per day before use.
Rechargeable equipment batteries	Charge.	After use as required.
Sampling accessories	Periodic maintenance performed and recorded in equipment maintenance log.	As required.

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9 Analytical Procedures

To accomplish the objectives of the RI/FS, laboratory analyses will be performed for PCB congeners, dissolved organic carbon by persulfate oxidation, total suspended solids, and weight-loss-on-ignition. A summary of the methodologies to be employed is included in Table 9-1. Table 9-2 provides a listing of the PCB congeners to be analyzed for in Phase 2B. The detection limit goals for the PCB congeners and conventional parameters are defined in Section 5.

Table 9-1

Analytical Procedures

Parameter	Method	Appendix
PCB Congeners	GC/ECD	A-2, A-3, A-4
PCB Congener Confirmation	GC/ITD	A-2, A-3, A-5
Total Suspended Solids	EPA Method 160.2[1]	--
Weight-Loss-on-Ignition	Project Specific[2]	B-2
Dissolved Organic Carbon by Persulfate Oxidation	Project Specific[2]	B-1

1 U.S. EPA, "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020 EMSL, Cincinnati, OH, Revised March 1983. Published method will be modified slightly by use of 0.7 μm filter.

2 Project-specific method utilized by Lamont Doherty Geological Observatory and Rensselaer Polytechnic Institute.

Table 9-2
PCB Congeners

BZ#	PCB Congener
1	2-Chlorobiphenyl
2	3-Chlorobiphenyl
3	4-Chlorobiphenyl
4	2,2'-Dichlorobiphenyl
5	2,3-Dichlorobiphenyl
6	2,3'-Dichlorobiphenyl
7	2,4'-Dichlorobiphenyl
8	2,4'-Dichlorobiphenyl
9	2,5-Dichlorobiphenyl
10	2,6-Dichlorobiphenyl
12	3,4-Dichlorobiphenyl
15	4,4'-Dichlorobiphenyl
16	2,2',3-Trichlorobiphenyl
18	2,2',5-Trichlorobiphenyl
19	2,2',6-Trichlorobiphenyl
22	2,3,4'-Trichlorobiphenyl
25	2,3',4-Trichlorobiphenyl
26	2,3',5-Trichlorobiphenyl
27	2,3',6-Trichlorobiphenyl
28	2,4,4'-Trichlorobiphenyl
29	2,4,5-Trichlorobiphenyl
31	2,4',5-Trichlorobiphenyl
37	3,4,4'-Trichlorobiphenyl
40	2,2',3,3'-Tetrachlorobiphenyl
41	2,2',3,4-Tetrachlorobiphenyl
44	2,2',3,5'-Tetrachlorobiphenyl
47	2,2',4,4'-Tetrachlorobiphenyl
49	2,2',4,5'-Tetrachlorobiphenyl
52	2,2',5,5'-Tetrachlorobiphenyl
53	2,2',5,6'-Tetrachlorobiphenyl
56	2,3,3',4'-Tetrachlorobiphenyl
66	2,3',4,4'-Tetrachlorobiphenyl
70	2,3',4',5-Tetrachlorobiphenyl
75	2,4,4',6-Tetrachlorobiphenyl
77	3,3',4,4'-Tetrachlorobiphenyl
82	2,2',3,3',4-Pentachlorobiphenyl
83	2,2',3,3',5-Pentachlorobiphenyl
84	2,2',3,3',6-Pentachlorobiphenyl
85	2,2',3,4,4'-Pentachlorobiphenyl
87	2,2',3,4,5'-Pentachlorobiphenyl
91	2,2',3,4',6-Pentachlorobiphenyl
92	2,2',3,5,5'-Pentachlorobiphenyl
95	2,2',3,5',6-Pentachlorobiphenyl
97	2,2',3',4,5-Pentachlorobiphenyl
99	2,2',4,4',5-Pentachlorobiphenyl
101	2,2',4,5,5'-Pentachlorobiphenyl
105	2,3,3',4,4'-Pentachlorobiphenyl
107	2,3,3',4',5-Pentachlorobiphenyl
115	2,3,4,4',6-Pentachlorobiphenyl
118	2,3',4,4',5-Pentachlorobiphenyl
119	2,3',4,4',6-Pentachlorobiphenyl
122	2',3,3',4,5-Pentachlorobiphenyl
123	2',3,4,4',5-Pentachlorobiphenyl
126	3,3',4,4',5-Pentachlorobiphenyl
128	2,2',3,3',4,4'-Hexachlorobiphenyl
129	2,2',3,3',4,5-Hexachlorobiphenyl
136	2,2',3,3',6,6'-Hexachlorobiphenyl
137	2,2',3,4,4',5-Hexachlorobiphenyl
138	2,2',3,4,4',5'-Hexachlorobiphenyl
141	2,2',3,4,5,5'-Hexachlorobiphenyl
149	2,2',3,4',5',6-Hexachlorobiphenyl
151	2,2',3,5,5',6-Hexachlorobiphenyl
153	2,2',4,4',5,5'-Hexachlorobiphenyl
157	2,3,3',4,4',5'-Hexachlorobiphenyl
158	2,3,3',4,4',6-Hexachlorobiphenyl
167	2,3',4,4',5,5'-Hexachlorobiphenyl
170	2,2',3,3',4,4',5-Heptachlorobiphenyl
171	2,2',3,3',4,4',6-Heptachlorobiphenyl
177	2,2',3,3',4',5,6-Heptachlorobiphenyl
180	2,2',3,4,4',5,5'-Heptachlorobiphenyl
183	2,2',3,4,4',5',6-Heptachlorobiphenyl
185	2,2',3,4,5,5',6-Heptachlorobiphenyl
187	2,2',3,4',5,5',6-Heptachlorobiphenyl
189	2,3,3',4,4',5,5'-Heptachlorobiphenyl
190	2,3,3',4,4',5,6-Heptachlorobiphenyl
191	2,3,3',4,4',5',6-Heptachlorobiphenyl
193	2,3,3',4',5,5',6-Heptachlorobiphenyl
194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl
195	2,2',3,3',4,4',5,6-Octachlorobiphenyl
196	2,2',3,3',4,4',5',6-Octachlorobiphenyl
198	2,2',3,3',4,5,5',6-Octachlorobiphenyl
199	2,2',3,3',4,5,6,6'-Octachlorobiphenyl
200	2,2',3,3',4,5',6,6'-Octachlorobiphenyl
201	2,2',3,3',4',5,5',6-Octachlorobiphenyl
202	2,2',3,3',5,5',6,6'-Octachlorobiphenyl
205	2,3,3',4,4',5,5',6-Octachlorobiphenyl
206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl
207	2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl
208	2,2',3,3',4,,5,5',6,6'-Nonachlorobiphenyl
209	Decachlorobiphenyl

Note: BZ# = Ballschmitter and Zell System.

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10 Data Reduction, Validation, and Reporting

Protocols for data reduction and reporting are summarized in Figure 10-1. Field data will be entered into bound serialized notebooks. Originals of field notebooks, chain-of-custody forms, field data sheets, and laboratory reports will be filed and stored. These documents are tracked during a periodic inventory during audits performed under the direction of the TAMS/Gradient Quality Assurance (QA) Officer of the project, Dr. A. Dallas Wait. See Section 18 for definitions of abbreviations employed in the following section.

10.1 Data Reduction

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. Data will be generated and reduced following protocols specified in the appendices to this SAP/QAPjP or in laboratory SOPs for standard methods. Each analyst will review the quality of his/her work, based on an established set of guidelines. This will constitute the "primary review". The analyst will review the data package to verify that:

- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- Analytical results are correct and complete (including calculations);
- QC sample results are within established control limits;
- Blanks are within established control limits;
- Special sample preparation and analytical requirements have been met;
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented; holding times are documented, etc.);
- All corrections on raw data and any generated forms are made with a single-line cross-out and initialed and dated by the analyst.

The primary analyst will initial and date all documents generated by him/her. A "secondary review" of the data generated by the primary analyst will be performed. This will entail a spot-check of the above listed items.

Any errors found will trigger a 100% check of all data included in that item. The secondary reviewer will initial and date all reviewed documents.

Data reduction will include provision of periodically updated summary tables containing the following information to the Quality Assurance Officer:

- Collection Date
- Sample Identification Number
- Sample Description
- Sample Location
- Laboratory Number
- Parameter
- Concentration and units
- Analysis Date

Interpretation of raw data and calculation of results are signed and dated by the laboratory scientist performing the data reduction on the data report forms. Another scientist, often the laboratory manager, must verify the results and sign the data before it is released. Additionally, a member of the laboratory QA staff should perform an audit of 5% of the data generated.

10.2 Data Validation

Data validation is the process of reviewing data and accepting, qualifying, or rejecting it on the basis of sound criteria. The data generated during this program will be validated according to established guidelines in this SAP/QAPjP and the USEPA Region II data validation SOPs (USEPA, 1992). Given the non-standard methods contained in this SAP/QAPjP, the data validation approach must consist of a systematic review of the results, associated quality control methods and results, and the supporting data using professional judgment in areas not specifically addressed by USEPA guidelines. For the PCB congener analyses, a specific data validation SOP has been developed to address the low level detection limit requirements of GC/ECD (Appendix A-6) and the congener confirmation by GC/ITD (Appendix A-7). For other analytical parameters, the data validation will address the following:

- Completeness:

The data package for each Sample Delivery Group (SDG) must include the following items.

1. Traffic Report and Chain-of-Custody (COC) forms.

2. Case narrative listing non-compliance issues.

3. Cover page; tabulated QC results and sample results. At a minimum these will include: tabulated sample concentrations; MS/MSD/MD results with % recoveries and % RPD per analyte; all blanks tabulated (method blanks and laboratory blanks); LCSs with % recoveries; ICVs and CCVs with % recoveries; surrogate recoveries; and method detection limits. Additional details are listed in Section 10.3 entitled Data Reporting.

4. Raw data supporting all analyses.

5. Raw data supporting all standardizations, calibrations and QC samples.

6. Preparation or extraction logs for all tests, matrices and samples.

7. Laboratory and sampling team IDs are consistent and can be tracked throughout data.

8. Holding times are documented.

- Accuracy

Review of laboratory control samples (LCS) and matrix spiked (MS) samples (where applicable) to determine accuracy based on % recovery of a known spiked compound.

$$Spike\ recovery = \frac{(spiked\ sample\ value - sample\ value)}{spike\ added} \times 100\%$$

- Precision

Review of laboratory matrix duplicates (MD), matrix spike duplicates (MSD) and field duplicates (FD) where applicable. Based on relative percent difference (RPD) between the duplicate values.

$$RPD = \frac{sample\ value - duplicate\ value}{\left(\frac{sample\ value + duplicate\ value}{2}\right)} \times 100\%$$

For MS/MSD precision, the sample and duplicate values are both spiked sample results.

- Detection Limits

Review of data reporting limits with SAP/QAPjP specific requirements.

- Blank Contamination

Review of all blanks (field blanks, method/preparation blanks, laboratory analytical blanks) to assess validity of the data based on criteria set for blank levels in the SAP/QAPjP.

The data acceptance limits for LCS, MS/MSD, MD, all blanks, ICVs and CCVs are defined within the methods and this SAP/QAPjP.

It is important that quantitation limits be kept as low as possible for PCB congener analyses. It is expected that the quantitation limits defined in Section 5 will be met. Precision and accuracy requirements have been defined in Section 5. Guidelines for acceptable surrogate standard recoveries, spike recoveries and RPD of duplicates in

both waters and particulates have been defined in this SAP/QAPjP based on EPA Region II and National Functional Guidelines criteria and technical references as listed in Section 17. These guidelines will be used in evaluating data quality.

In addition to the above directives, protocols from the following documents will be used to validate the inorganic and organic data:

1. CLP Organics Data Review and Preliminary Review. January 1992, SOP No. HW-6, Rev. #8. U.S. EPA Region II.

2. Evaluation of Inorganic Data for the Contract Laboratory Program. January 1992, SOP No. HW-2, Rev. XI, US EPA Region II.

3. Data validation SOPs, Appendices A-6 and A-7.

4. National Functional Guidelines for Organic Data Review.

10.3 Data Reporting

For PCB congener data, appropriate CLP forms (modified as necessary) for pesticide/PCB reporting should be used where applicable. The specific deliverables are defined in the applicable SOP. Sufficient QC, supporting, and raw data shall be provided so that validation of the data can be performed. In general, data reports for each sample analyzed will include the following information:

- Final analyte concentration.
- Laboratory sample ID#, field sample ID#, location.
- Percent solids (for sediment samples).
- Final volume of extract or prepared sample.
- Preparation or extraction and analysis dates for holding time verifications.
- Calibration information, including (where applicable):
  - calibration curve

- correlation coefficient, and
- concentration response data of the calibration check standards.

- Results of the second column chromatography check including chromatograms (PCB analysis only).
- Amount of surrogate spiked and percent recovery of each surrogate.
- For matrix spike samples, the amount spiked and % recovery of each compound or analyte spiked.
- For matrix duplicate or spike duplicate samples, % RPD calculated for each compound or analyte.
- For laboratory control samples, or matrix spike blanks true values and % recovery of each analyte quantitated.
- Blank results for method blanks, field blanks and laboratory analytical blanks.
- Raw data and preparation and extraction logs must include:
  - analyst initials and date
  - initial and final sample volumes or weights
  - sample description artifacts (e.g. stones, standing water in sediment samples, color)
  - amount and concentration of stock spike solutions added to MS/MSD or LCS samples
  - Vendor or Lot Number identification for all initial and continuing check samples and true value concentrations of these check standards (ICV, CCV, etc.).
- All raw data analysis printouts and logs must include:
  - analyst initials and date
  - Model Number and type of instrument used for analysis
  - conditions of instrument (e.g. wavelength for colormetric analyses, retention times for GC, etc.)
  - time of start of analysis, time for all QC samples, time of end of analysis
  - Method Number or SOP reference
  - dilutions performed and amount of sample analyzed or injected
  - calibration standards labeled and time recorded
  - QC samples and blanks clearly labeled.

FIGURE 10-1
PROTOCOLS FOR DATA REDUCTION AND REPORTING:
CONVENTIONAL RAS/SAS LABS

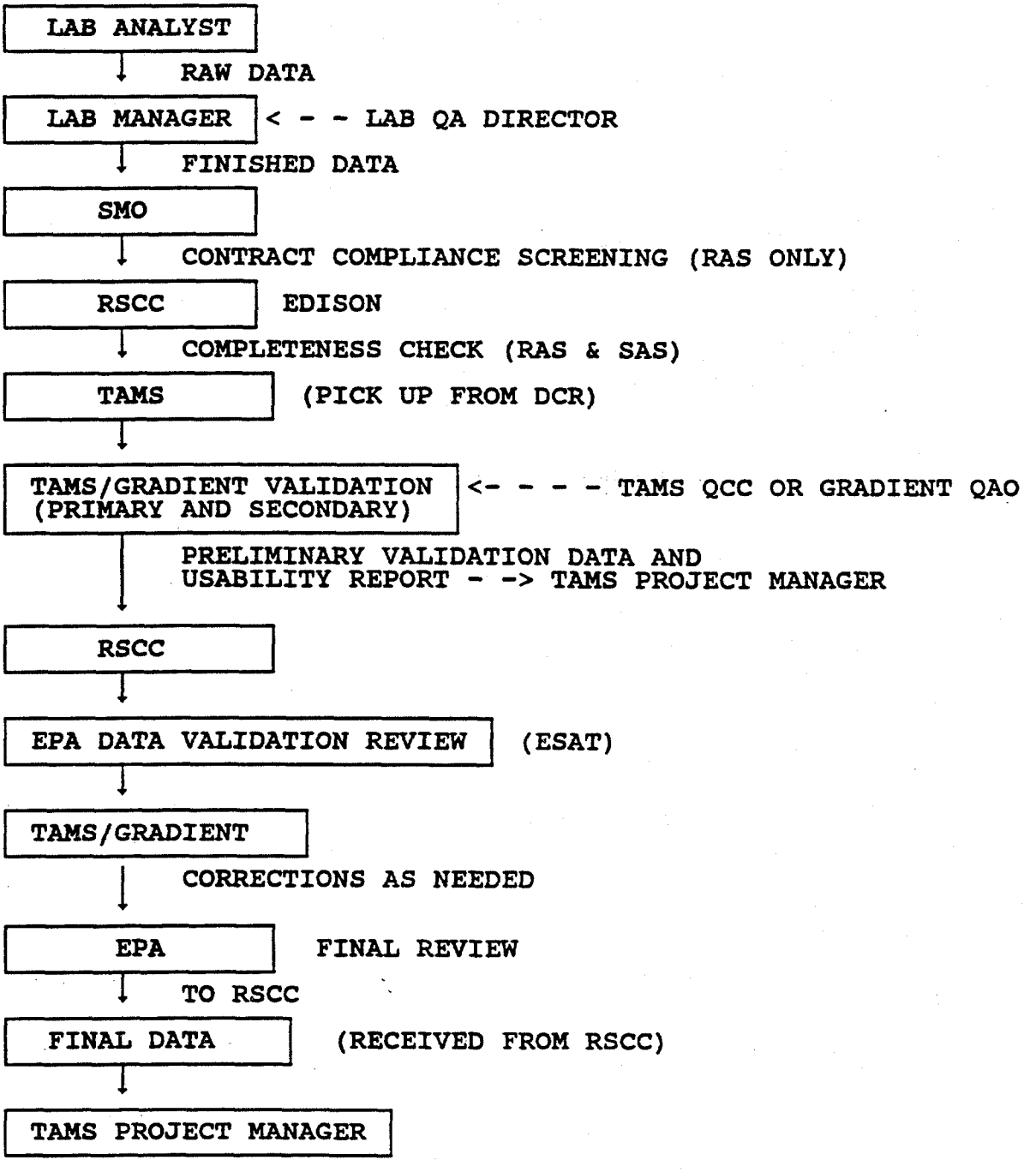
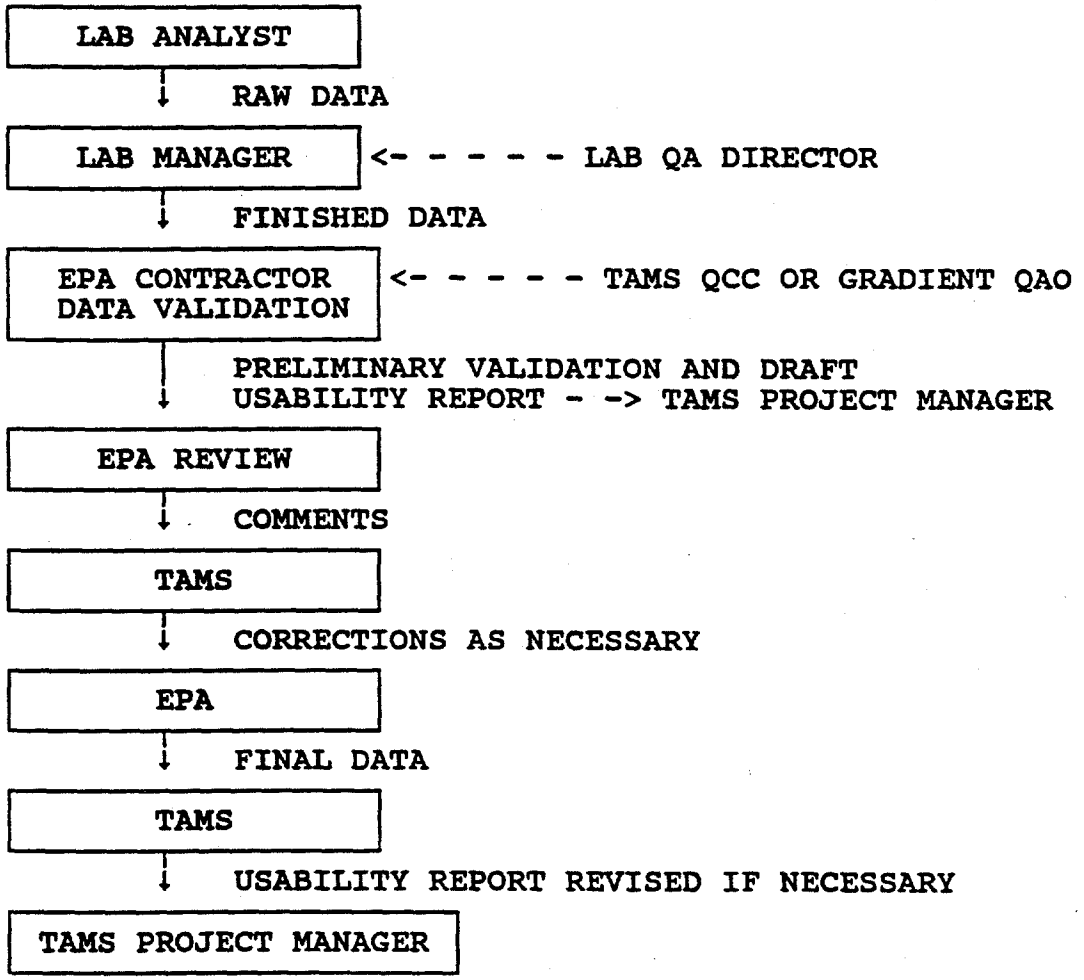


FIGURE 10-2
PROTOCOLS FOR DATA REDUCTION AND REPORTING:
LABS CONTRACTED TO TAMS



11 Internal Quality Control Checks

The type and frequency of field, matrix, and laboratory specific Quality Control (QC) checks are summarized in Tables 11-1, 11-2, and 11-3. Method SOPs must be referenced for more detailed information (in Appendices A and B of this SAP/QAPjP).

11.1 Field Quality Control Checks

Quality control checks will be instituted as part of the sampling program. Field quality control samples to be collected include field blanks, field duplicates, and analyte-free water blanks.

11.1.1 Field Blanks

Field blanks will be collected by the field sampling team and analyzed by the laboratory in order to assess possible contamination from sampling equipment or field operations. A field blank consists of deionized analyte-free water passed through the field sampling apparatus, preserved as a sample, and submitted to the laboratory for analysis. The frequency of field blanks will be a minimum of one per decontamination event per matrix for each type of sampling equipment. Field blanks will be collected and analyzed for PCB congeners, TSS/WLOI, and DOC.

The analyte-free water for PCB congener field blanks will be provided by the analytical laboratory. PCB congener field blanks will be sixteen liter samples. Analyte-free water for other field blank analyses will be procured by TAMS from a commercial source, and will be the same size as the corresponding field sample. If TSS is not detected in a particular field blank, WLOI analysis will not be required on that blank.

11.1.2 Field Duplicates

Field duplicate samples (field replicates) will be obtained to assess the adequacy (precision) of overall sampling and handling procedures as well as the representativeness of the samples. A minimum 5% frequency for field duplicate pairs (i.e., one pair per 20 samples) will be taken and analyzed per matrix per analysis. Field duplicates will be analyzed for all project parameters (PCB congeners; TSS; WLOI; and DOC).

11.1.3 Analyte-Free Water Blanks

The analyte-free water procured by TAMS from commercial sources is analyzed on a regular basis for TCL/TAL analytes. For the flow-averaged water sampling investigation, analysis for TOC and TSS will also be included.

No analyte-free water blank will be submitted for PCB congener analysis. The quality of the laboratory-provided analyte-free water will be assessed by analysis of field blanks for PCB congeners and the laboratory analysis of method blanks.

11.2 Matrix Specific QC

Matrix Spike (MS)/Matrix Spike Duplicate (MSD)/Matrix Duplicate (MD) Samples: a MS/MSD pair will be performed for PCB congener analysis at the frequency of one per 20 samples (5%) per matrix or per SDG, whichever is more frequent. All other parameters will have a matrix duplicate (MD) only. Since samples for TSS and WLOI analyses cannot be split in the laboratory, MD analyses for these parameters will be performed on identified (not blind) replicates submitted from the field. MD analyses are in addition to blind field duplicate analyses. See Table 11-1 for laboratory QC summary per parameter.

The purpose of the MS is to assess matrix effects on percent recovery of the compound or analyte. MS data can also be used to measure accuracy of the method with the caution that specific matrix effects may obscure the results. MSD measures the same features as MS, with the additional information on relative percent difference (RPD) between the MS and MSD. This is a measure of the precision of the method. The MD measures precision for all analytes other than the PCB congeners in this program. The % RPD between the sample and MD concentrations are determined and compared to the criteria specified in individual SOPs and in Table 5-1.

Field measurement QC checks are summarized in Table 11-2. Criteria for field measurements are given in Table 5-2.

11.3 Laboratory Quality Control Checks

Table 11-3 lists the frequency of laboratory QC checks. Accuracy and precision criteria for LCS, MS/MSD/MD are given in Section 5. Method-specific criteria for continuing calibration checks, blanks, and other criteria are presented in the individual SOPs in the Appendices. For the flow-averaged samples, each 15-day event will generate four environmental samples, which will constitute one Sample Delivery Group (SDG). Since 32 discrete grab samples for TSS (and an undetermined quantity for WLOI, depending on TSS concentration) will be collected for each 15-day event, there will be two SDGs for this parameter. At a minimum, the following items will be included as laboratory QC:

- Method Blanks
  These blank samples are prepared in the laboratory and are analyzed in order to assess possible laboratory contamination during the preparation or extraction procedure. The method or preparation blank must be analyzed at a frequency of one per matrix per parameter and per each batch of 20 samples or per SDG, whichever is more frequent.

- Analytical Blanks
  Analytical blanks are the routine analysis of laboratory reagent-grade water during the analytical process to assess contamination and instrument drift. These are equivalent to instrument blanks (CCB) for PCB congener analysis.

- Performance Evaluation (PE) Samples
  PE samples will be submitted blind to each laboratory performing PCB congener analyses every 3 to 6 months. A sediment PE sample which is a NIST Standard Reference Material (SRM) is available and will be used as the PE sample for the particulate matrix. If an aqueous PCB congener SRM can be obtained, aqueous PE samples will also be submitted as appropriate.

The laboratory will maintain its own internal QC program, as summarized below.

- For each parameter and each matrix, minimum of one method (procedural) blank in every batch of 20 samples or SDG, whichever is more frequent, will be analyzed to detect

contamination.

- For each parameter and matrix (as applicable; see Tables 11-1 and 11-3), a minimum of one laboratory control sample (LCS) per batch or every 20 samples, whichever is more frequent. The LCS will be used to access laboratory performance of the method. LCS for water samples will consist of distilled deionized water spiked with the analyte of interest. The matrix spike blank for PCB congener analysis will serve the function of the LCS and will be spiked with the same standard spike mix as used for MS Samples. Where available for other analyses, the LCS will consist of an independently prepared sample of known or accepted true concentration. However, for most analyses in this program, the ICV is equivalent to the LCS for laboratory method evaluations. For TOC, the ICV is defined as requiring the same preparation and analysis methods as for a sample. For this reason, the ICV can be interpreted as an LCS since it fulfills the requirements of a "blank spike sample" or "laboratory control sample".

- For PCB congeners, a minimum of one MS/MSD pair per matrix per batch of 20 samples or per SDG, whichever is more frequent, to assess accuracy and determine matrix effects.

- Surrogate standards to estimate recoveries and to account for sample-to-sample variation as required in the PCB method.

- For PCB congener analysis, 5-point multilevel initial calibrations of instruments to establish calibration curves. For other parameters, calibrations that require linear regressions to define the curve must have correlation coefficient (r) values ≥ 0.995. These other calibrations must consist, at a minimum, of 4 standards and one blank.

- Continuing calibration check every 5 samples for PCB congeners. For other parameters, calibration checks every 10 or 20 sample analyses (where applicable).

- Initial calibration checks or verification (ICV) performed immediately following calibration to determine accuracy of the daily calibration curve as compared to a separate source check standard. (Traceability of the ICV solution to an EPA or NBS [NIST] standard solution is

recommended.)

- All PCB samples will be analyzed on a secondary capillary column for PCB congener confirmation.

- Approximately 10% of the particulate samples and 5% of the waters (dissolved) analyzed for PCB congeners will require additional confirmation by GC/ITD (Appendix A-5). The GC/ITD analyses will be performed with the same capillary columns used for the GC/ECD analyses, and will employ similar congener standard mixes. The GC/ITD analyses are intended to confirm congener identification. In addition, quantitative comparability studies between GC/ECD and GC/ITD will be conducted. Quantitative deviations in the results of the two methods should be less than 50 percent.

TABLE 11-1
LABORATORY QUALITY CONTROL SUMMARY

	Quality Control Parameters									
Laboratory Parameters	**Method**	**ICV**	**ICB**	**CCV**	**CCB**	**MB**	**MSB (LCS)**	**MS**	**MSD**	**MD**
PCB congeners - GC/ECD	P1			X	X	X	X	X	X	
PCB Congeners - GC/ITD	P2				X					
Dissolved organic carbon	P3	X	X	X	X	X				X
Total suspended solids	E1	X	X			X				X[1]
Weight loss-on-ignition	P4	X				X				X[1]

[1] These analyses cannot be split into multiple analytical aliquots in the laboratory. Therefore, the matrix duplicate analyses for these parameters will be performed on an identified (not blind) field replicate submitted for matrix duplicate analysis. (Blind field duplicates will also be submitted.)

Methods:

P1 = Project specific method for PCB congeners by gas chromatography/electron capture detection (Appendix A-4)
P2 = Project-specific method for PCB congener confirmation gas chromatography/ion trap detection (Appendix A-5)
P3 = Project-specific method for organic carbon by persulfate oxidation (Appendix B-1)
P4 = Project-specific method for weight loss on ignition (Appendix B-2)
E1 = EPA method 160.2

Notes:

In some cases, the ICV may equal the CCV or the LCS and ICB may equal CCB. For PCB congeners by GC/ECD, the MSB serves as the LCS. See method SOP and section 11 for specific requirements. The quality control parameters are defined in Table 11-3.

TABLE 11-2
FIELD QUALITY CONTROL SUMMARY

	Quality Control Parameters				
Field Parameters	**Matrix**	**Calibration**	**ICV**	**CCV**	**MD**
pH	Water	X		X	X
Conductivity	Water	X			
Temperature	Water	X			
Dissolved Oxygen	Water	X			X

Methods:

See field method SOPs in Appendix C.

Notes:

pH: CCV = pH 7 buffer solution

The quality control parameters are defined in Table 11-3.

Table 11-3

QC Frequency Summary

For tests that specify the following QC, this table summarizes the frequency requirements. See method SOPs and Tables 11-1 and 11-2 for applicable QC per parameter.

QC	Frequency
Initial Calibration Verification Check (ICV)	1 per analytical run immediately following calibration (all parameters except PCB congeners)
Initial Calibration Blank (ICB)	1 per analytical run immediately following the ICV where applicable (all parameters except PCB congeners)
Continuing Calibration Verification Check (CCV)	Every 12 hours during analytical run for PCB congeners (= continuing calibration check), every 10 to 20 samples for other parameters (see SOPs)
Continuing Calibration Blank (CCB)	Every 12 hours for PCB congeners (instrument blanks for GC/ECD). Every 10 to 20 samples immediately following CCV where applicable (see SOPs)
Laboratory Control Sample (LCS)	1 per 20 or SDG whichever is more frequent (for PCB congener analysis only; the matrix spike blank = LCS; for other analyses the LCS = ICV)
Matrix Spike (MS)	1 per 20 or SDG whichever is more frequent
Matrix Spike Blank (MSB)	1 per 20 or SDG, whichever is more frequent, for GC/ECD PCB congener analysis only; MSB = LCS.
Matrix Spike Duplicate (MSD)	1 per 20 or SDG whichever is more frequent (PCB congener analysis only)
Matrix Duplicate (MD)	1 per 20 or SDG whichever is more frequent (all parameters expect PCB congeners)
Method (Preparation) Blank (MB)	1 per 20 or SDG whichever is more frequent
Field Blank (FB)	1 per matrix per parameter per decontamination event, where applicable
Field Duplicate (FD)	1 per matrix per parameter per 20 samples taken: minimum frequency of 5%
Performance Evaluation (PE)	1 every 3 to 6 months for each available matrix (non-aqueous and aqueous) for each laboratory analyzing PCB congeners.

12 Performance and System Audits and Frequency

Audits of the field sampling team and of the laboratories performing work in support of this program will be performed under the direction of the Quality Assurance Officer. At least one on-site audit will be performed during sampling at the PCB analytical facility and at the RPI laboratory.

Audits during the program will be performed at a frequency to satisfy the QA Officer that the analyses are progressing within QC limits set forth in this SAP/QAPjP and following specific method SOPs documented herein. Frequency of laboratory audits may occur at biweekly intervals or greater, tapering off to monthly or bimonthly as the program proceeds.

12.1 Field Audits

Specific elements of the on-site audit will include the verification of the following items:

- Completeness and accuracy of sample Chain-of-Custody (COC) forms.
- Completeness and accuracy of sample identification labels.
- Completeness and accuracy of field notebooks.
- Following proper Health & Safety procedures as outlined in the Health & Safety Plan for this program.
- Following specific decontamination procedures as outlined in Section 6.4 of this SAP/QAPjP and delineated in the Sampling Plan for this program.
- Following specific collection, preparation, preservation and storage procedures outlined in Section 6.2 and 6.3 of this SAP/QAPjP.
- Following specific calibration and analytical procedures for field parameters as outlined in field parameter SOPs in Appendices C-2 through C-4 of this SAP/QAPjP.
- Following handling and shipping procedures outlined in Section 6.5 and Appendix C-1 of this SAP/QAPjP.

Appendix D-1 is an example of a Field Sampling Audit Checklist.

12.2 Laboratory Audits

The TAMS-contracted laboratories involved in analyses for this program will be audited under the direction of the QA Officer at the frequency listed above. Due to the special requirements associated with many of the non-routine methods of this investigation, emphasis in these audits will focus on evaluating the technical adequacy of the analyses as it pertains to the data quality objectives. In particular, the laboratory performing the PCB congener analyses will be expected to be experienced with the methods to employ sound scientific judgment as necessary.

An example checklist for laboratory audits pertaining to routine technical requirements and document control systems is provided in Appendix D-2. Items will be addressed as applicable to the specific method being reviewed during the audit. The following items, at a minimum, will be addressed:

- Sample flow through lab and internal sample tracking
- Chain-of-Custody
- Sample storage
- Sample preparation/extraction and analysis including:
    - SOPs
    - Logbooks or benchsheets for all preparation procedures of samples, calibration standards, QC standards/check samples, blanks
    - Logbooks or benchsheets for all analytical procedures for samples, calibrations, QC checks, matrix QC samples, blanks
    - All above documentation must include:
        - analyst initials and date
        - single-line cross-out for corrections, initials and date
        - units recorded
        - method reference number or SOP reference

- Consistency with the laboratory's QA Program Plan and the project-specific requirements of this SAP/QAPjP.
- QC samples documentation inclusive of items above and for all blanks, calibrations, calibration verification check samples, laboratory control samples, spikes, duplicates, spike duplicates, surrogates, control charts (were applicable)
- Data file storage including hard copy of all data, other media (disk, tape, etc)
- Laboratory safety procedures
- Laboratory QA procedure including internal audits, corrective action forms, QC control charts

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13 Preventive Maintenance Procedures and Schedules

Field sampling personnel will be familiar with the field calibration, operation and maintenance of the equipment, and will perform the prescribed field operating procedures outlined in the Operation and Field Manuals accompanying the respective instruments and the SOPs attached in the Appendices C-2 through C-4.

Laboratory staff will be familiar with the maintenance requirements of the instrumentation they employ. This familiarity is the result of technical education, specialized courses and laboratory experience. Wherever possible, the laboratory will maintain a complete inventory of replacement parts needed for preventive maintenance and spare parts that routinely need replacement. It is the laboratory's responsibility to maintain maintenance log books for each instrument used in this program. These will be checked during the laboratory audits and must be kept current with information on routine and non-routine maintenance procedures.

Preventive maintenance schedules for analytical instrumentation will be specific to the laboratory's instrument manufacturer's specifications. Maintenance procedures and schedules will be outlined in the laboratory's SOPs and will be strictly adhered to for this program.

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14 Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness

The following are specific definitions for precision, accuracy and completeness. Also, see Section 5 for further information.

14.1 Precision

Precision is frequently determined by the comparison of replicates, where replicates result from an original sample that has been split for identical analyses. Relative percent difference and standard deviation of a sample are commonly used in estimating precision.

Analyses performed in this program will have a measure of precision in terms of matrix duplicates, matrix spike duplicates, field duplicates or in the case of some analyses, laboratory duplicates. See specific method SOPs (Appendices A and B) and Section 5 for further details.

14.1.1 Relative Percent Difference

For laboratory (matrix) duplicates (samples that result when an original sample has been split into two for identical analyses) and field duplicates, the relative percent difference (RPD) between the two samples will be used to evaluate precision.

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

where: D_1 = first sample value
D_2 = second sample value (duplicate)

14.1.2 Standard Deviation

Standard deviation(s) is calculated as:

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2}$$

where a quantity x_i (e.g., a concentration) is measured n times with a mean $\bar{x}$.

The relative standard deviation, RSD (or sample coefficient of variation, CV), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates.

$$RSD = 100\ (s/\bar{x})$$

or

$$CV = 100\ (s/\bar{x})$$

where: RSD = relative standard deviation, or
CV = coefficient of variation
s = standard deviation
$\bar{x}$ = mean

14.2 Accuracy

The determination of accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy is assessed by calibration verification, LCS or matrix spike sample analyses, and analysis of other samples whose true value is known to the laboratory. In addition, analytical accuracy will

be assessed by submission of performance evaluation (PE) samples to the laboratory (whose true value is unknown to the laboratory). Accuracy may be calculated in terms of bias as follows:

$$Bias = \bar{X} - T$$

$$\% \ Bias = \frac{100(\bar{X} - T)}{T}$$

where: $\bar{X}$ = average observed value of measurement
T = "true" value

Accuracy will also be calculated in terms of the recovery of spiked samples as in the case of matrix spike samples for this program. Surrogate recovery is also calculated for PCB analyses as an indicator of the accuracy of the method on a particular sample.

$$\% \ Recovery = 100 \left[\frac{X}{T}\right]$$

14.3 Completeness

Determining whether a data base is complete or incomplete may be quite difficult. To be considered complete, the data set must contain the required QC check analyses verifying precision and accuracy for the analytical protocol. Less obvious is whether the data are sufficient to achieve the goals of the project. Data are reviewed in terms of project goals in order to determine if the data base is sufficient. Following data validation, the % completeness can be obtained as the following calculation:

$$\% \ Completeness = \frac{valid \ data \ obtained}{total \ data \ planned} \times 100$$

It should be noted that a number of factors may result in obtaining less than 100% of the planned data, including field conditions (e.g., a planned sampling location was inaccessible), sample handling or shipping (e.g., sample bottles broken in transit), and analytical deficiencies (serious QC and problems resulting in the data being unusable [rejected]).

15 Corrective Action

The acceptance limits for the sampling and analyses to be conducted in this program have been defined in Sections 5, 8, 9 and 11. The corrective actions are likely to be immediate in nature and most often will be implemented by the field sampling personnel or laboratory analyst. The corrective action will usually involve recalculation, repreparation, reanalysis, or repetition of a sample run.

15.1 Immediate Corrective Action

Specific QC procedures and checklists are designed to help analysts detect the need for corrective action. In addition, a scientist's experience will be valuable in alerting the operator to suspicious data or malfunctioning equipment.

If a corrective action is taken as part of normal operating procedures, the collection of poor quality data will be avoided. Instrument and equipment malfunctions are amenable to this type of corrective action, and the QC procedures will include troubleshooting guides and corrective action suggestions. The actions taken will be noted in field or laboratory notebooks or benchsheets and a memorandum issued to the QA Officer within one day of the corrective action. No other formal documentation will be provided, unless further corrective action is necessary. These on-the-spot corrective actions are an everyday part of the QA/QC system. Corrective action during the field sampling portion of the program is most often a result of equipment failure or an operator error (omission), and may require repeating a sampling event. Operator error is best avoided by having field crew members audit each other's work before and after a test. It is the responsibility of the Field Operations Leader to maintain adherence to the specified QC procedures.

Laboratory personnel will be alerted that corrective actions may be necessary if:

- QC data are outside the acceptable windows for precision and accuracy;
- Blanks contain contaminants above acceptable levels (> MDLs);
- Undesirable trends are detected in spike recoveries or the relative percent difference between duplicates;

- There are unusual changes in detection limits;
- Deficiencies are detected by the laboratory QA Director during internal audits or from the QA Officer during program audits;
- Inquiries concerning data quality are received from the client (USEPA).

Corrective action procedures are often handled at the bench level by the analyst who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, and instrument sensitivity. If the problem persists or cannot be identified, the matter is referred to the Laboratory QA Manager or Director. Once resolved, full documentation of the corrective action procedure is filed with the laboratory QA department.

15.2 Long-Term Corrective Action

The need for long-term corrective action may be identified by standard QC procedures, control charts, performance, or system audits. Any quality problem which cannot be solved by immediate corrective action falls into the long-term category. The Laboratory QA Director shall see that the condition is reported to a person responsible for correcting it, who is part of a closed-loop action and follow-up plan.

The essential steps in the closed-loop corrective action system will include:

- Identification and definition of the problem.
- Delegation of responsibility for investigating the problem.
- Investigation and determination of the cause of the problem.
- Determination of a corrective action to eliminate the problem.
- Delegation and acceptance of responsibility for implementing the corrective action.
- Establishment of effectiveness of the corrective action and its implementation.
- Verification that the corrective action has eliminated the problem.

Documentation of the problem is important to the system. A Corrective Action Request Form (shown on Figure 15-1), or equivalent, will be completed by the person finding the quality problem. This form identifies the problem, possible causes and the person responsible for action on the problem. The responsible person may be an

analyst, Field Operations Leader, or the QC Director. If no person is identified as responsible for action, the QC Director will investigate the situation and determine who is responsible in each case.

The Corrective Action Request Form includes a description of the corrective action planned, the date it was taken, and space for follow-up. The QC Director will check to verify that initial action has been taken, appears effective, and at an appropriate later date will, check again to verify that the problem has been fully solved. The QC Director will receive a copy of all Corrective Action forms, and will enter them in the Corrective Action Log. This permanent record will aid the QC Director in follow-up action and this log will be reviewed by the QA Officer during program audits.

Figure 15-1
Corrective Action Request Form

Corrective Action Request Form No. _____

Originator _____ Date _____

Person Responsible for Replying _____ Contract Involved _____

Description of problem and when identified: _____

State cause of problem if known or suspected: _____

Sequence of Corrective Action: (If no responsible person is identified, notify QA Manager immediately. Submit all CA forms to QA Manager for initial approval of CA.)

State Date, Person, and Action Planned:

CA Initially Approved By: _____ Date _____
Follow-up Dates: _____
Final CA Approval By: _____ Date _____

Information copies to:
RESPONSIBLE PERSON/DEPARTMENT QC COORDINATOR: _____
QA MANAGER: _____
DEPARTMENT MANAGER: _____

16 Quality Assurance Reports to Management

The Quality Assurance Officer will issue reports pertaining to all quality assurance assessments and issues which occur during the project. The reports will include, as appropriate, the results of the field and laboratory audits, document audits, significant quality problems discovered, and any necessary corrective action procedures. A data quality assessment and data usability report, based on all the samples and the data validation reports, will be incorporated into the final report.

Reports for field and laboratory audits will be submitted to the TAMS project manager within 10 days following the audit. Serious deficiencies will be reported within one day of the audit with corrective actions identified.

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18 List of Abbreviations for QA/QC Criteria

CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification (Continuing Calibration Check) Sample
CLP	Contract Laboratory Program
COC	Chain-of-Custody
DCR	Document Control Room
ESAT	Environmental Services Assistance Team (EPA Contractor)
FB	Field Blank
FD	Field Duplicate Sample
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification (Initial Calibration Check) Sample
LCS	Laboratory Control Sample
MB	Method (Preparation/Extraction) Blank
MD	Matrix Duplicate Sample
MDL	Method Detection Limit
MS	Matrix Spike Sample
MSB	Matrix Spike Blank
MSD	Matrix Spike Duplicate Sample
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
PE	Performance Evaluation
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPjP	Quality Assurance Project Plan
QC	Quality Control
QCC	Quality Control Coordinator
RAS	Routine Analytical Services
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan

SAS	Special Analytical Services
SDG	Sample Delivery Group
SMO	Sample Management Office
SOP	Standard Operating Procedure
TAL	Target Analyte List (Inorganics)
TCL	Target Compound List (Organics)

A

APPENDIX A

PCB EXTRACTION, ANALYSIS, AND DATA VALIDATION

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Appendix A-2
Extraction and Cleanup of Large Volume Water Samples for PCB Congener Analysis

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Appendix A-3
Extraction and Cleanup of Sediments and Particulates for PCB Congener Analysis

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Appendix A-4
Congener Specific Determination of Polychlorinated Biphenyls (PCBs) in Hexane Extracts by Capillary Column Gas Chromatography

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Appendix A-5
Congener Specific Determination of Polychlorinated Biphenyls (PCBs) in Hexane Extracts by Capillary Column Gas Chromatography/Ion Detector (GC/ITD) - Confirmation Analyses

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Appendix A-6
Standard Operating Procedure Data Validation for Congener Specific Determination of Polychlorinated Biphenyls (PCBs) by Gas Chromatography/Electron Capture Detector (GC/ECD)

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Appendix A-7
Standard Operating Procedures Data Validation for Congener Specific Determination of Polychlorinated Biphenyls (PCBs) by Gas Chromatography/Ion Trap Detector (GC/ITD)

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B

APPENDIX B

CONVENTIONAL AND OTHER LAB ANALYSES

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Appendix B-1
Organic Carbon - Persulfate Oxidation Method

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APPENDIX B-1

MEASUREMENT OF DISSOLVED ORGANIC CARBON IN WATER SAMPLES BY PERSULFATE DIGESTION

1.0 Scope and Application

1.1 This method describes a procedure for the determination of dissolved organic carbon (DOC) in field-filtered water samples. The procedure is designed to measure natural water levels of dissolved organic carbon on a per unit volume basis. "Dissolved" organic carbon is defined operationally; that is, the total organic carbon of a field-filtered sample is defined in this method as dissolved organic carbon.

2.0 Summary of Method

2.1 Samples are field-filtered and acidified (preserved) with sulfuric acid. Then, 0.1 g potassium persulfate is added to a sample aliquot and purged of all CO_2 with a stream of CO_2-free helium. The ampule is sealed and heated to 90° C for 4 hours. A portion of the sample is then withdrawn, mixed with an equal volume of helium, and shaken. The helium, now containing sample CO_2, is injected into a gas chromatograph and the CO_2 level is measured by a thermal conductivity detector.

2.2 Method interferences may result from the loss of volatile organic compounds during the initial stripping of CO_2 from the sample. Additional interferences may occur from contaminated laboratory glassware if care is not taken with low-level samples.

3.0 Apparatus and Materials

3.1 Field-filtering Apparatus

The apparatus for field-filtering consists of a stainless steel filter holder using a glass fiber filter or Gelman membrane filter. The specific dimensions and filtering procdedure are described in the applicable Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPjP).

3.2 Gas Chromatograph

An analytical system complete with gas chromatograph and all required accessories including syringes, analytical columns, thermal conductivity detector and a strip-chart or an electronic recording device. The chromatograph must have a loop injection system to provide a reproducible injection volume.

3.3 Hot Water Bath

A hot water bath is needed to maintain samples at 90° C. Temperature control must be accurate to ± 5°C.

4.0 Reagents

4.1 Sample Preservation - samples will be preserved as described in the SAP/QAPjP. The temperature of the shipping container shall be checked upon receipt by the laboratory. The pH of the samples shall be checked and recorded prior to analysis.

4.2 Helium - CO_2-free for stripping samples of CO_2 before beginning digestion.

4.3 Potassium persulfate - Reagent grade

4.4 Calibration standards

Glucose solution in five different concentrations (e.g., 100, 200, 500, 1000, and 2000 umol/l, corresponding to 1.2, 2.4, 6.0, 12, and 24 mg organic carbon/liter) made with high purity distilled deionized water.

4.5 Distilled, deionized water, 18 Mohm purity, minimum dissolved organic carbon content (preferably $\leq$0.05 mg/l organic carbon).

5.0 Initial Calibration

5.1 The gas chromatographic system must be initially calibrated using the five standard solutions described in Section 4. All standard solutions are prepared for analysis on the gas chromatograph following the procedure described in Section 6 with the exclusion of the filtration step. Prior to the analysis of these standards, the laboratory must determine the retention time of CO_2 on the instrument.

5.2 Tabulate peak height or area responses against concentration for each standard.

5.3 Fit a first or second order regression equation to the calibration data. No higher order of regression is permitted. The correlation coefficient must be ≥ 0.995. A plot of the calibration curve and standards must be supplied with the sample results.

5.4 The full range calibration must be done for every group of 20 samples or less.

6.0 Procedure

6.1 Field Procedure

The field procedure for collecting and filtering the sample is described in the SAP/QAPjP.

6.2 Laboratory Procedure

6.2.1 Measure out 24 ml of sample from the sample bottle, add 0.1 gm of potassium persulfate and place in a 25 ml ampule.

6.2.2 Strip CO_2 from sample for 3 minutes using a thin diameter tube and CO_2-free helium. The helium flow should be about 0.5 cc/minute.

6.2.3 Remove tube and immediately flame-seal the ampule.

6.2.4 Place sample in hot water bath at 90° C for at least four hours.

6.2.5 Break ampule and remove about 18 cc of sample using a 30 cc plastic syringe fitted with a syringe valve. Expel all air bubbles and some water until 15 cc of sample are left in syringe.

6.2.6 Fill syringe to 30 cc with CO_2-free helium, yielding a syringe half filled with water and half with helium. Close syringe valve and shake vigorously for two minutes. Place syringe in shaker bath at 25° C for at least five minutes.

6.2.7 Inject gas into gas chromatograph. Inject about 8 cc or sufficient gas to purge injection loop.

6.2.8 Inject gas filled loop into gas chromatograph and record response.

7.0 Calculations

7.1 Calculate the mass of dissolved carbon in each of the samples using the response curve generated in Section 5.

7.2 Calculate the relative percent difference (RPD) for duplicate pairs:

$$RPD = \frac{Sample\ Level - Sample\ Duplicate\ Level}{\left(\frac{-Sample\ Level + Sample\ Duplicate\ Level}{2}\right)} \times 100\%$$

Compare % recovery of ICV and CCV to "true" value. The % recovery criterion is 90-110%.

8.0 Precision and Accuracy

8.1 The precision of the method is expected to be ≤25% (RPD) on matrix (laboratory) duplicates.

8.2 The achievable detection limit is approximately 0.25 mg/l (based on 5 times the expected method blank level of 0.05 mg/l organic carbon).

8.3 The anticipated accuracy of the measurement is 90% to 110%, based on ICV and CCV recoveries.

9.0 Quality Control

9.1 Method Blanks

Since most distilled deionized water contains some dissolved organic carbon, a reliable method blank can only be generated using a previously digested distilled, deionized water blank. Blanks should be ≤0.05 mg/l organic carbon.

9.1.1 To prepare a method blank, two previously analyzed distilled deionized water blanks are required. Approximately 12 ml of solution are taken from each water blank and combined in a clean, unused ample for a total of 24 ml.

9.1.2 0.1 gm of potassium persulfate are added.

9.1.3 The solution in bubbled with CO_2-free helium for at least 3 minutes at 0.5 cc/sec using a thin tube.

9.1.4 The tube is removed and the ampule quickly sealed with a flame.

9.1.5 The remainder of the preparation and analysis follows steps 6.2.4 to 6.2.8.

9.1.6 One method blank will be run for every twenty samples or for every sample delivery group, whichever is more frequent.

9.2 Calibration

A five point calibration will be performed for every twenty samples or for every sample delivery group, whichever is more frequent.

9.3 Laboratory Calibration Verification

In addition to the five point calibration to be run with every sample delivery group, an independent laboratory calibration standard (ICV) will be run. This standard will be derived from a separate source or vendor than that for the five calibration standards. The measured value of the standard must recover within 90% to 110% calculated as:

$$\%\ recovery = \frac{measured\ value\ of\ ICV}{true\ value\ of\ ICV\ standard} \times 100\%$$

9.4 Continuing Calibration Verification

The mid-range standard (CCV) will be repeated during the analytical process after every 10 samples and at the end of the day's operation to check instrument drift. This standard must agree to within 10% of the true value. If this is not achieved, all samples run since the last time the containing calibration check was in control or since the last five point calibration must be rerun. The laboratory must first re-establish control by recalibrating the instrument and rerunning the ICV prior to continuing sample analyses.

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Appendix B-2
Weight Loss on Ignition

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Appendix B-2

WEIGHT LOSS-ON-IGNITION ANALYSIS

1.0 Scope and Application

1.1 This method describes the procedure for the determination of weight loss on ignition by combustion of sediment or of non-filterable suspended solids retained by a glass fiber filter.

2.0 Method Summary

2.1 A small amount of sediment or a glass fiber filter containing suspended solids weighed after drying (20 to 80 mg) is heated to 375°C in air for 16 to 18 hours to remove all organic material and reweighed.

2.2 Method interferences may be caused by loss of water by dehydroxylation of clays in the sediments if the heating temperature is brought to 500°C.

3.0 Apparatus and Materials

3.1 Precision Balance - The balance must be capable of weighing to 0.1 mg for samples weighing several grams.

3.2 Autoclave or Muffle Furnace - This unit must be capable of controlling the combustion temperature to $\pm$ 10°C at 375°C.

3.3 For suspended solids, the filter must consist of glass fiber. Membrane filters are not acceptable since they will combust.

3.4 Cleaning procedures: crucibles will be rinsed with distilled/deionized water and heated to dryness at 110°C.

4.0 Reagents

4.1 Internal laboratory standard (LCS) of Hudson River sediment, containing about 5% organic materials (5% loss on ignition).

5.0 Initial Calibration

5.1 Balances must be calibrated daily using "S" class weights. The balance calibration is checked using the internal standard at least once per day. The balance is allowed to return to zero between each sample weighing.

5.2 The internal laboratory standard (LCS) is run once for each group of 20 samples or sample delivery group, whichever is more frequent.

6.0 Methodology

6.1 Sediments

6.1.1 A weighed sediment sample of about 0.2 to 0.5 gm is dried to a constant weight in an incubator oven at 110°C for approximately 2 hours. Historical data show that 2 hours is sufficient to provide a constant weight (i.e., successive weighings meet a relative percent difference (RPD) criterion of $<0.5\%$). The laboratory must demonstrate the adequacy of the drying by demonstrating that this criterion is met for successive weighings at least 15 minutes apart for at least one sample in each drying batch.

6.1.2 Sample is placed in a muffle furnace or autoclave at 375°C for 14 to 16 hours.

6.1.3 Sample is allowed to cool in a drying cabinet and then reweighed.

6.2 Filter (Suspended Solids) Sample

6.2.1 The mass of suspended matter on the filter must be previously determined (e.g., by EPA method 160.2) before determining weight loss-on-ignition. This mass is equal to the total suspended solids (TSS). This is accomplished by using a pre-fired (500°C) pre-weighed glass fiber filter column, filtering the water sample, and drying to a constant weight (following step 6.1.1 above). The TSS

(mass of suspended matter) is the difference between the preweighed filter weight and the dried filter weight.

6.2.2 Follow steps 6.1.2 and 6.1.3 as defined for sediments.

7.0 Calculations - Sediments and Filters

7.1 Calculate the relative percent difference (RPD) for the dried sample as:

$$RPD = \frac{Wt\ 1 - Wt\ 2}{\left(\frac{Wt\ 1 + Wt\ 2}{2}\right)} \times 100\%$$

where: Wt 1 and Wt 2 are the sample weights at two successive weighings separated by at least 4 hours. These weighings are taken prior to baking at 375°C.

7.2 Calculate the weight loss on ignition (WLOI) as follows:

$$LOI = \frac{Wt\ 2 - Wt\ 3}{Wt\ 2} \times 100\%$$

where: Wt 3 is the weight of the sample after heating for 14 to 16 hours at 375°. Wt 2 is the last weight of the sample taken prior to heating at 375°C.

7.3 Calculate the RPD for sample duplicates and laboratory standards as follows:

$$RPD = \frac{LOI\ 1 - LOI\ 2}{\left(\frac{LOI\ 1 + LOI\ 2}{2}\right)} \times 100\%$$

where: LOI 1 = the loss on ignition calculated for the first sample analysis or, in the case of the laboratory standard, the established loss on ignition value.

LOI 2 = the loss on ignition calculated for the duplicate sample or standard analysis.

8.0 Precision and Accuracy

The precision criterion, based on matrix (laboratory) duplicate results, is $\leq$25% RPD, or $\leq$0.5 mg for a sample containing $\leq$2.0 mg total suspended solids. Accuracy criterion of the recovery of the internal laboratory standard (LCS) is 90-110% recovery.

Due to the limitations of the weighing measurement, the minimum detection limit is considered to be 0.5 mg. On a sample containing 50 mg of suspended matter, this represents 1.0% WLOI.

9.0 Quality Control

9.1 Method Blank - A method blank is analyzed with each group of 20 samples or sample delivery group (SDG), whichever is more frequent. The method blank consists of an empty crucible (for sediments) or a new glass fiber filter (for suspended matter). The WLOI must be $\leq$0.4 mg.

9.2 The laboratory control standard (LCS) is equivalent to an initial calibration check (ICV) for this analysis, and is run with each sample batch must be compared to a previously established weight loss on ignition value for the standard. The criteria for the % recovery is 90% to 110%.

9.3 If either of these criteria are exceeded, the entire sample batch must be reanalyzed from new sample material. For filter samples, reanalysis cannot be performed as the entire sample is used in the preparation. Problems should be reported in the data package narrative and to the Project Quality Assurance Officer.

9.4 Sample Duplicates - One laboratory (matrix) duplicate analysis will be run for at least every 20 samples or SDG, whichever is more frequent. Laboratory duplicates will be performed on additional sample volume submitted from the field and designated for this purpose. The WLOI duplicate will be performed on the same filter used for the TSS laboratory duplicate. Laboratory duplicates must meet an RPD of 20%, or $\pm$0.5 mg for TSS $\leq$2.0 mg. If this criterion is not met a third sample portion should be determined, where possible. All three values must be reported.

C

APPENDIX C

FIELD PROCEDURES

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Appendix C-1
Sample Packaging and Shipping SOP

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Appendix C-2
pH - Field Measurement SOP for Waters

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Appendix C-3
Dissolved Oxygen - Field Measurement SOP for Waters

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Appendix C-4
Salinity, Conductivity, and Temperature - Field Measurement SOP for Waters

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APPENDIX D

QUALITY ASSURANCE AUDITS

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D-1
Field Quality Assurance Audits

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D-2
Laboratory Quality Assurance Audits

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