



**Hudson River PCBs Site  
Baseline Monitoring Report  
Data Summary Report for 2006**

*Prepared for:*

**General Electric Company  
Albany, NY**

*Prepared by:*

**Quantitative Environmental Analysis, LLC  
Glens Falls, NY**

*In conjunction with:*

**Environmental Standards, Inc.  
Valley Forge, PA**

**March 30, 2007**



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***VIA FEDERAL EXPRESS***

March 30, 2007

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***Re: 2006 Baseline Monitoring Program (BMP) Data Summary Report***

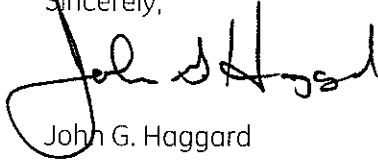
Dear Sir or Madam:

Attached is the 2006 Baseline Monitoring Program (BMP) Data Summary Report for your review and approval. This report documents the field and laboratory work performed under the BMP between January and December 2006, presents the results, and assesses overall data quality.

March 30, 2007  
Page 2

If you have any questions do not hesitate to contact Bob Gibson or me.

Sincerely,

A handwritten signature in black ink, appearing to read "John G. Haggard". The signature is stylized with a large, looping initial "J" and a long, sweeping horizontal stroke.

John G. Haggard

JGH/bg  
Enclosure

cc: Robert Gibson, GE  
Sheri Moreno, GE  
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Cathy Beebe, GE  
John Connolly, QEA

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**Job Number:**

**GENbmp:131**

**March 30, 2007**

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- Appendix C. BMP project database (on CD-ROM).
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- Appendix E. USGS flow and NRCC meteorological data (on CD-ROM).
- Appendix F. List of samples validated for each method (on CD-ROM).
- Appendix G. Data validation reports (on CD-ROM).

### **List of Acronyms**

ASTM	American Society for Testing and Materials
BMP	Baseline Monitoring Program
BMPSD	Baseline Monitoring Program Scoping Document
CAM	Corrective Action Memorandums
cfs	Cubic Feet per Second
COC	Chain-of-Custody
CRQL	Contract Required Quantitation Limit
DO	Dissolved Oxygen
DOC	Dissolved Organic Carbon
DQOs	Data Quality Objectives
DSR	Data Summary Report
DVM	Data Verification Module
EDDs	Electronic Data Deliverables

EDI	Equal Discharge Increment
EMPCs	Estimated Maximum Possible Concentrations
ESI	Environmental Standards, Inc.
GE	General Electric Company
GPS	Global Positioning System
HRM	Hudson River Mile
HRRM	Hudson Reference Material
LCSs	Laboratory Control Spikes
LDs	Laboratory Duplicates
MADIS	Multiple Aliquot Depth Integrating Sampler
MDL	Method Detection Limit
mGBM	Modified Green Bay Congener Method
MS	Matrix Spikes
MSDs	Matrix Spike Duplicates
NEA	Northeast Analytical Laboratory, Inc.
NRCC	Northeast Regional Climate Center at Cornell University
NYSDEC	New York State Department of Environmental Conservation
PCBs	Polychlorinated Biphenyls
PCRDMP	Post-Construction Remnant Deposit Monitoring Program
PE Sample	Performance Evaluation Sample
POC	Particulate Organic Carbon
Pseudo-TOT	Pseudo-Time-of-Travel
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QDQ	Qualitative Data Quality
QEA	Quantitative Environmental Analysis, LLC
RD AOC	Hudson River Remedial Design and Cost Recovery
RM	River Mile
ROD	Record of Decision
RPD	Relative Percent Difference
SDGs	Sample Delivery Groups

SOP	Standard Operating Procedures
STL	Severn Trent Laboratories
TAL	Target Analyte List
TID	Thompson Island Dam
TKN	Total Kjeldahl Nitrogen
TOT	Time of Travel
TSS	Total Suspended Solids
EPA	United States Environmental Protection Agency
USGS	United States Geologic Survey

## **SECTION 1 INTRODUCTION**

### **1.1 BACKGROUND**

This annual data summary report (DSR) for the 2006 Baseline Monitoring Program (BMP) has been prepared on behalf of General Electric Company (GE) by Quantitative Environmental Analysis, LLC (QEA), in conjunction with Environmental Standards, Inc. (ESI). The purpose of this report is to document the field and laboratory work performed to complete the BMP in 2006, report the data, and to present the results of the associated data quality assessment.

The 2006 BMP was conducted under the Administrative Order on Consent for Hudson River Remedial Design and Cost Recovery (RD AOC), effective August 18, 2003 (Index No.CERCLA-02-2003-2027; United States Environmental Protection Agency [EPA] and GE 2003), as part of the remedial design to implement the February 2002 Record of Decision (ROD) for the Hudson River PCBs Site issued by EPA (EPA 2002). The overall goals and scope of the BMP are defined in the Baseline Monitoring Program Scoping Document (BMPSD; QEA 2003), which was attached to the RD AOC. The BMP entails the routine collection and analysis of water and fish samples, as well as the performance of several special studies to support the remedial design. The methods and data quality objectives (DQOs) of the program are detailed in the BMP Quality Assurance Project Plan (QAPP; QEA and ESI 2004), which was approved by EPA on May 21, 2004.

### **1.2 REPORT OBJECTIVES**

The objective of this DSR is to document the BMP activities completed in 2006 and to present the resulting data. Data interpretation efforts in this report are limited to assessing data quality and usability. The QAPP specifies that the annual report is to contain the following information: “The DSR will fully document the calendar year’s work including a summary of

the work performed, a tabulation of results, field notes, processing data, chain-of-custody (COC) forms (information is incorporated into lab analytical data packages), copies of laboratory audits, data validation results, copies of laboratory reports, and a CD version of the project database”.

### **1.3 REPORT ORGANIZATION**

This report is divided into ten sections that summarize the BMP field and lab activities for 2006. Section 1 includes the introduction and objectives. Section 2 provides a summary of the methods followed during the BMP water program, fish program, and special studies. Section 3 summarizes the quality assurance/quality control (QA/QC) methods used for the 2006 BMP. Sections 4, 5, and 6 present the results of the water program, fish program, and special studies, respectively. Section 7 presents the results of the portion of the BMP that was performed to satisfy the requirements of the Post-Construction Remnant Deposit Monitoring Program (PCRDMP; QEA 2000). Section 8 presents an assessment of data quality. Section 9 gives an overall summary of 2006 BMP activities. Section 10 contains the references. A total of seven appendices are included that provide documentation for the various field, laboratory, and data validation activities.

## **SECTION 2 METHODS**

### **2.1 ROUTINE WATER SAMPLING PROGRAM**

Sampling was conducted at stations in the Upper and Lower Hudson River and in the Mohawk River (Figure 2-1). A summary of the sampling schedule is presented in Table 2-1. Sampling was performed weekly at the following six stations:

- Bakers Falls (River Mile [RM] 197.0);
- Rogers Island (RM 194.2);
- Thompson Island (RM 187.5);
- Schuylerville (RM 181.4);
- Stillwater (RM 168.4); and
- Waterford (RM 156).

Bakers Falls and Rogers Island are considered background monitoring stations. The remaining stations will be far-field monitoring stations during dredging. Data collected at these stations during remediation will be assessed to determine achievement of the resuspension performance standards (RPS; EPA 2004).

#### **2.1.1 Sample Collection Procedures**

Water column samples were collected on a weekly basis in accordance with the standard operating procedures (SOP) specified in Appendix 1 of the QAPP (SOP for Weekly Water Column Sampling; ESI and QEA 2004). Modifications to the sampling procedures were implemented based on recommendations made in corrective action memorandums (CAMs). A discussion of the CAMs is presented in Section 2.1.1.1.

Samples collected at Bakers Falls were taken at the approximate centroid of the river cross section from the downstream side of Bakers Falls Bridge (County Rt. 27 Bridge). At Rogers Island, aliquots were collected from the center of the east and west channels using a boat. These aliquots were combined to form a composite sample using a volume ratio consistent with the flow ratio in the east and the west channel (60:40). To satisfy the lower polychlorinated biphenyl (PCB) analytical sensitivity requirements at these stations, 8 L of water were collected for each PCB sample from Bakers Falls and Rogers Island.

The remaining routine water sampling stations were sampled at either five or six substations located along transects across the river cross section. Sampling at Thompson Island was conducted from a boat at six EDI stations placed along a transect located downstream of the southern tip of the island (Figure 2-2). Transect sampling at Schuylerville was conducted from a boat along the upstream side of the Rt. 29 Bridge at six EDI stations (Figure 2-3). Transect sampling at Stillwater was conducted from a boat along the upstream side of County Rt. 125 Bridge at five EDI stations to the west of the entrance to Lock 4 land cut (Figure 2-4). Transect sampling at Waterford was conducted from a boat along the upstream side of the Rt. 4 Bridge at five EDI locations (Figure 2-5).

The samples for each of these stations consisted of a single composite made up of depth-integrated aliquots collected at each substation. A variable speed bridge or boat mounted crane (Figure 2-6) was used to lower a custom-designed multiple aliquot depth integrating sampler (MADIS; Figure 2-7) containing twelve or sixteen 500-ml glass sample collection vessels, depending on sample volume requirements. The speed and distance that the sampler was lowered was adjusted according to water depth at each substation to allow collection of an appropriate sample volume. Each sample collection vessel was outfitted with a special cap with a sampling nozzle and air vent. The sampler was lowered through the water column to within approximately two feet of the river bottom and then retrieved, such that a depth-integrated sample aliquot was collected. Sample aliquots were retained when the volumes varied no more than  $\pm 20\%$  of the target volume for the sampling location (e.g., 1/5 of the total sample volume for a transect with five EDI locations).

The entire sample volume collected from each substation along the transect was combined to generate a single composite sample for each parameter or related set of parameters at each monitoring station. This process was repeated at each transect substation using the same sample collection vessels. Sample containers used to collect the PCB sample(s) at each station were retained and transported to the laboratory along with the water sample(s). At the laboratory, the empty sample container was rinsed with hexane and the hexane rinsate was combined with the sample extract such that any residual PCBs from the sampling container were included in the PCB analysis.

#### ***2.1.1.1 Corrective Action Memoranda (CAM) Issued***

Five CAMs were prepared and presented to EPA in 2006 (Appendix A). These memos documented proposed changes to the sampling procedures. The CAMs proposed the following modifications to the sampling program:

- CAM005 (submitted April 27, 2006):
  - Proposed discontinuation of nutrient analysis. The nutrient samples collected during 2004 and 2005 indicated little variability and fulfilled the requirement of the DQO to establish baseline conditions. This CAM was implemented based on verbal agreement with EPA.
- CAM006 (submitted May 1, 2006):
  - Proposed discontinuation of sampling at the historical stations. In accordance with verbal discussions with EPA, the Schuylerville station was discontinued in June 2006. At the request of EPA, additional analyses were performed for data collected from the Thompson Island Dam station. The results of these analyses were presented to EPA on August 10, 2006. Sampling has continued at this station pending input from EPA.
- CAM007 (submitted May 2, 2006):
  - Proposed reduction of the sampling frequency of sampling at Bakers Falls to monthly. This CAM has not yet been put in effect pending input from EPA regarding the implications of this modification on the requirements of the PCRDMP.
- CAM008 (submitted July 26, 2006):

- Provides an updated SOP for ICP/MS analysis of water samples by Method 200.8 and an SOP for hardness analysis. This CAM has been implemented.
- CAM009 (submitted May 11, 2006):
  - Updated SOP for the determination of total organic carbon in solids and water and the determination of particulate and dissolved total organic carbon in water. This CAM has been implemented.

#### **2.1.1.2 TAL Metals**

Samples were collected and analyzed for EPA target analyte list (TAL) metals in accordance with the water sampling and the Dissolved Metals SOPs (QAPP Appendices 1 and 44; QEA and ESI 2004). Upon completion of preparation of the composite sample at a transect location, a portion of that sample was designated for dissolved metals analysis was transported to a dedicated field laboratory facility for filtration. The sample was filtered through a 0.45  $\mu\text{m}$  filter using the “clean-hands/dirty hands” procedure described in Appendix 44 of the QAPP. The resulting filtrate was placed in an appropriate container, double-bagged, and placed in a cooler with ice prior to transport to the analytical laboratory.

#### **2.1.2 Field Parameters**

Water quality parameters were collected at mid-depth at each sampling location (centroid or EDI) using portable field instrumentation. This instrumentation consisted of a YSI 650 data logger and a YSI 6920 multiparameter sonde equipped with turbidity, pH, temperature, dissolved oxygen (DO), and conductivity probes. Instrument calibration and data collection procedures were conducted in accordance with the SOP (QAPP Appendix 2; QEA and ESI 2004).

#### **2.1.3 Waterford High Flow Sampling**

PCB, total suspended solids (TSS), particulate organic carbon (POC), and dissolved organic carbon (DOC) samples were collected at Waterford during seven high flow events in 2006. High flow conditions are defined as flow at the United States Geologic Survey (USGS)

gauging station at Fort Edward, NY (Station ID: 01327750) exceeding 15,000 cubic feet per second (cfs) or peak flow at Waterford expected to reach 22,500 cfs. Sampling was conducted at a centroid location from the Route 4 Bridge using the same methods described in Section 2.1.1.

The QAPP specifies that the timing of the high flow sample collection will be based on instantaneous flow obtained at the Fort Edward USGS gauging station. This procedure was followed whenever possible; however, access to the telephone gauge height was not available during a portion of 2006, preventing obtaining instantaneous data. During these times, sample collection times were estimated based on near-real time hydrographs published by USGS on the internet. At other times, flows were elevated at Waterford, but not at Fort Edward due to the influence of localized precipitation events in the drainage basin between Fort Edward and Waterford. These conditions prevented the use of the Fort Edward hydrograph to select sample collection times. Additionally, reliable flow data is not available during the navigational season from the Waterford gauge when the Lock 1 dam pool is filled. When potential high flow events were identified under these circumstances, flow at Waterford was estimated by combining flow provided by USGS for the Battenkill, Hoosick River, and the Hudson River at Fort Edward. The timing of sample collection has been modified for 2007 in accordance with discussions with EPA.

#### **2.1.4 Mohawk River Water Column Monitoring**

Mohawk River water samples were collected monthly during 2006 from the Rt. 32 Bridge at Cohoes and analyzed for PCBs and TSS. The Mohawk River was not sampled in January or February due to ice conditions. Sampling was conducted at five EDI locations (Figure 2-8) using the same methods described in Section 2.1.1.

#### **2.1.5 Lower Hudson Water Column Monitoring**

Sampling in the Lower Hudson River at Albany/Troy and Poughkeepsie was conducted monthly from May through November 2006. The samples were collected from a boat at a centroid location (defined as the approximate center of the channel; Figure 2-1). A single, depth

integrated sample was collected with the MADIS sampler. Due to the depth of the channel at the Poughkeepsie location (approximately 75 ft.), the MADIS sampler was only lowered through the uppermost 50 ft. of the water column.

## **2.2 SPECIAL STUDIES**

With the exception of the sampling performed at the historical Thompson Island Dam (TID) and Schuylerville monitoring stations described below, the special studies specified in the QAPP (QEA and ESI; 2004) were completed and discontinued in 2005.

### **2.2.1 Historical Stations**

To provide a means to compare between the historical record of PCB concentrations at Thompson Island Dam (TID) and Schuylerville, and PCB data collected under the BMP, the historical single point sampling locations at TID (TID-PRW2) and Schuylerville were sampled once per month concurrent with routine water column sampling using the historical sampling methods associated with the PCRDMP (PCRDMP; QEA 2000). This method involved lowering a Kemmerer bottle sampler into the water column to collect a sample. The method was repeated until sufficient volume was collected for all the parameters. PCB and TSS samples were collected monthly at Schuylerville, from March through May, when it was discontinued in accordance with CAM 006. Samples were collected at TID-PRW2 from April through November. Field parameters were also monitored at these stations.

### **2.2.2 Additional TSS Samples**

During May and June 2006, an additional sample was collected at TID and Schuylerville for TSS analysis each week. This resulted in the collection of eight additional samples from TID and nine from Schuylerville.

## **2.3 FISH PROGRAM**

The BMP fish program was initiated in the spring of 2004 in accordance with the QAPP (QEA and ESI 2004). In 2006, adult fish were sampled in the spring and yearling pumpkinseed and forage fish were sampled in late summer. Fish collection was targeted within five pools of the Hudson River:

- Feeder Dam Pool (one station).
- Thompson Island Pool (five stations).
- Northumberland Pool (four stations; one abandoned in 2004).
- Stillwater Pool (five stations).
- Albany/Troy (one station – below Federal Dam in spring; Albany turning basin in fall).

The spring and fall fish sampling transect locations are depicted in Figure 2-9 and Figure 2-10, respectively.

### **2.3.1 Spring Species**

Spring fish sampling occurred from May 30 through June 8, 2006 (Table 2-2). During sampling, adult species of black bass (largemouth and smallmouth bass), perch (yellow and white perch), and ictalurids (brown and yellow bullhead; white and channel catfish) were targeted from the 15 stations in the Upper Hudson River and one location in the Lower Hudson River (below Federal Dam in Troy). A total of 374 samples were collected from the spring sampling locations, corresponding to 126 individuals from the bass group, 122 from the bullhead group, and 126 from the perch group (Table 2-2). Collections of adult fish targeted the legal or edible size: >305, >200, >170, and >160 mm total length, for bass, bullhead/catfish, yellow perch, and white perch, respectively.

Twenty fish per species were targeted in the spring in the Feeder Dam Pool and at Albany/Troy. Occasionally, when obtaining the targeted number of fish was problematic, adjustments to the program were made in the field following agreement by EPA oversight

personnel (Ron Sloan of New York State Department of Environmental Conservation (NYSDEC). The targeted number of bullhead was not obtained from the landlocked section (ND2); two bullhead were obtained instead of the target of five. An additional yellow perch was retained from this station to compensate for fewer bullhead. Thirty fish per species were collected in the spring in Thompson Island Pool with ten individuals per species from the historical location behind Griffin Island (TD5) and five individuals per species from each of the four other stations. In Northumberland Pool, five individuals per species were collected from two stations (ND1, ND3) and ten individuals per species were collected from one station (ND5) to account for the lack of fish at ND4. At ND2, only two fish from the bullhead group were collected. Six fish from the perch group were kept due to the lack of bullhead at that station. Thirty fish per species were collected from Stillwater Pool with ten individuals per species from the historical location at Coveville (SW3), and five individuals per species at each of the four remaining stations (SW1, SW2, SW4, and SW5). Additional details are presented in Section 2.3.3.

### **2.3.2 Fall Species**

Collection of forage fish and pumpkinseed occurred from August 28 through August 30, 2006 (Table 2-3). Forage fish were collected as whole body composites and included spottail shiner, bluntnose minnow, spotfin shiner, fallfish, and mimic shiner (one species per composite), based on availability. A total of 50 composites were targeted from the stations sampled in the late summer (ten composites per pool; Table 2-3). Pumpkinseeds were captured from each pool and submitted as whole body individual samples. Pumpkinseeds were considered yearlings if they were between 70 and 150 mm total length, in accordance with the requirements in the QAPP (QEA and ESI 2004). However, based on field discussions with EPA oversight, attempts were made to limit collection of pumpkinseed between 70 and 130 mm total length.

Twenty pumpkinseeds were collected at both Albany/Troy and the Feeder Dam Pool. Three composites out of ten of forage fish were collected at Albany/Troy due to a paucity of fish. Thirty pumpkinseeds were collected in the late summer in Thompson Island Pool, with ten

individuals from the historical location across from Griffin Island (TD5) and five individuals per species from each of the four other stations. In Northumberland Pool, access was not available in the landlocked section (ND1, ND2) since the private ramp used in previous events was not in safe condition. Samples were collected from the two other locations in Northumberland Pool in sufficient quantity to achieve the targeted numbers for this pool (25 pumpkinseed, 10 forage composites). Ten pumpkinseeds were collected from ND3, and fifteen pumpkinseeds were collected from (ND5), to account for the lack of fish at the other stations. Five forage fish composites each were collected at ND3 and ND5 due to lack of fish at the other stations. Thirty pumpkinseeds were collected from Stillwater Pool with ten individuals from the historical location at Coveville (SW3) and five individuals at each of the four remaining stations (SW1, SW2, SW4, and SW5).

### **2.3.3 Sampling Methods**

Electroshocking was used to collect target species. The edible portions for humans and wildlife were monitored; fillets for bass, ictalurids, and perch; individual, whole body samples for pumpkinseed; and whole body composites for spottail shiners or other forage fish species.

Electrofishing was accomplished with an 18 ft. boat equipped with a variable output gas-powered DC generator. Conductivity and turbidity were measured at each location prior to sampling to assess conditions. Operating amperage was adjusted according to water conductivity to minimize injury; stunned fish were immediately removed from the electrical field using dip nets to minimize the duration of the shock. Fish were held in live-wells or buckets with frequent water changes during collection. Fish were sacrificed by a blow-to-the-head or by cervical dislocation.

Sampling methods were generally consistent with procedures outlined in the QAPP (QEA and ESI 2004) with a few exceptions:

- Filleting of adult individuals was conducted in the analytical laboratory to eliminate the need for decontamination materials in the field.

- Weighing adult fish to the nearest 0.1 gram was not feasible due to the activity of the fish and the slight sway of the boat. Fish weight was recorded to the nearest gram.
- During sampling below the Federal Dam in Albany (spring 2006), no bullhead were collected and channel catfish and white catfish were used as surrogates. These species were not listed as a surrogate for bullhead in the QAPP; however, a decision was made in the field (in 2004) with EPA oversight personnel (Ron Sloan of NYSDEC), that channel catfish and white catfish would be acceptable surrogates.
- During 2004 spring sampling of Northumberland Pool, several attempts were made to collect fish from ND4. The habitat in this area of the river is sparse with steep-sided banks and a sharp drop-off into the river. No fish were observed during shocking. A decision was made in the field, with the concurrence of EPA oversight (Ron Sloan of NYSDEC) to abandon this location and collect an additional five individuals per species from ND5.
- During 2006 spring sampling of Northumberland Pool, only two bullhead were captured at ND2. Several attempts were made to collect the additional three species. A decision was made in the field with EPA oversight (Ron Sloan of NYSDEC) to suspend sampling and process the fish that were collected.
- During late summer sampling of Northumberland Pool, conditions for access into the landlocked section were not deemed safe so these two locations were not sampled. A decision was made in the field with EPA oversight (Ron Sloan of NYSDEC) to collect all of the fish from ND3 and ND5.
- During late summer sampling of the Albany turning basin, only three composites of forage fish were captured. A decision was made in the field with EPA oversight (Ron Sloan of NYSDEC) after a few hours of effort, to suspend sampling and process the fish that were collected.
- Based on discussion with EPA oversight (Ron Sloan, NYSDEC), pumpkinseed between 70 and 130 mm total length were targeted in consideration of the variability in the sizes and ages of pumpkinseed. The data for these 2007 pumpkinseeds and the pumpkinseeds collected during previous BMP sampling events provide data on PCB levels in fish that can be used to establish baseline conditions and to evaluate changes and system recovery

trends resulting from remediation; thereby satisfying the DQOs of the BMP fish collection and analysis.

Adult fish were collected along transects at each station during spring 2006. Transects were approximately 200 to 2,000 meters in length and were located parallel to the shoreline in water approximately one to three meters deep (Figure 2-9).

Fish were collected in late summer 2006 generally along the same transects sampled in the spring. Transects at a few stations were modified based on historic NYSDEC yearling pumpkinseed locations that were in slightly different areas than adult fish locations. Transects were approximately 200 to 1,000 meters in length and were located parallel to the shoreline in water approximately one to three meters deep (Figure 2-10).

Fish were handled according to SOPs developed by NYSDEC (NYSDEC 2000). Measurements were made as soon as possible following collection, with calibrated instruments. For each specimen, the date of collection, a unique identification number or code, the location, including coordinates, genus and species, total length in millimeters (to nearest mm), weight in grams (to nearest 1.0 gram), sex (done in the analytical laboratory during processing), and method of collection were recorded in the BMP fish field database. Each sample was then wrapped in clean aluminum foil (shiny side out), placed in a labeled plastic resealable storage bag, and kept on ice following data processing. The same information was also collected for composited fish, including number of individuals within the composite. Observed external abnormalities were also noted in the field database. COC forms were generated after data were entered into the field database and samples were kept on ice and delivered by courier to Northeast Analytical Laboratory, Inc. (NEA). Samples were processed by experienced personnel at the laboratory and prepared tissues (standard fillets or whole bodies) were frozen at a temperature below -18°C until analyzed. Fish samples were analyzed within the one-year holding time.

## **2.4 POST-CONSTRUCTION REMNANT DEPOSIT MONITORING**

Monitoring of the Hudson River in the vicinity of the Remnant Deposits is required by the PCRDMF Consent Decree (Consent Decree 1990), and includes weekly sample collection at Bakers Falls and Rogers Island. The routine monitoring conducted for the BMP at these stations satisfies the requirements of the PCRDMF; therefore, this DSR will satisfy the reporting requirements of the PCRDMF. Preparation of a specific PCRDMF annual summary report has been discontinued.

## **2.5 ANALYTICAL PROGRAM**

The BMP involved analysis of water and fish samples for chemical and physical parameters.

### **2.5.1 Water Program**

The routine measurements on the Upper Hudson water column samples included congener-specific PCBs, TSS, POC, and DOC. Congener-specific PCBs were quantified by single, whole water extraction.

Congener-specific PCBs, TSS, POC, and DOC were measured during the Waterford High Flow Sampling. Congener-specific PCBs and TSS were measured at the Lower Hudson water column monitoring locations.

Congener-specific PCB analysis of water samples was performed by NEA using the modified Green Bay Congener Method (mGBM) described in Appendix 9 of the QAPP (QEA and ESI 2004). Extraction and analysis techniques for congener-specific PCBs in Hudson River water were customized based on whether sampling stations require lower detection limit methods. The procedures employed were modifications to existing methods to improve sensitivity and/or to take advantage of current extraction technology. Brief descriptions of the

extraction and analytical methods for routine (1 L) and large-volume (8 L) samples are described in Sections B4.1.1 and B4.1.2 of the QAPP.

NEA analyzed 1 L water samples for TSS following the standard EPA protocol for the analysis of suspended sediment (Appendix 18 of the QAPP – *SOP for the Determination of Suspended Solids by EPA Method 160.2*; QEA and ESI; 2004) with modifications to be consistent with the American Society for Testing and Materials (ASTM) *D 3977-97 Standard Test Methods for Determining Sediment Concentration in Water Samples, Test Method B – Filtration* as described in Section 4.1.2 of the QAPP.

POC and DOC analyses were also performed by NEA using in-house method NE128\_03 as described in Appendix 19 of the QAPP (QEA and ESI 2004). TAL metals and hardness were analyzed by Severn Trent Laboratories (STL; Pittsburgh, PA) following the SOPs in Appendices 14 through 17 in the QAPP. TAL metals were analyzed by EPA Method 200.8, with the exception of mercury, which was analyzed by EPA Method 245.1. Hardness was analyzed by EPA Method 130.2.

## **2.5.2 Fish Program**

Fish were prepared for contaminant analyses following collection according to the *SOP for Annual Fish Sampling* (QAPP Appendix 21; QEA and ESI 2004). Fish samples were analyzed by NEA for total PCBs according to a modification of the EPA Method 8082 Aroclor Sum Method (NEA SOP 148, Revision 4; Appendix 25 of the QAPP; QEA and ESI 2004). Additionally, fish samples were analyzed by NEA to determine the lipid content according to the methods outlined in NEA SOP 158, Revision 3 (Appendix 24 of the QAPP). The mGBM (NEA SOP 133, Revision 1; Appendix 26 of the QAPP) was performed by NEA on 10% of the total number of fish samples.

Prior to analysis, fish tissue, either whole body or fillet, was homogenized following the methods outlined in NEA SOP 132 (Appendix 22 of the QAPP, QEA and ESI 2004). Extraction

and cleanup of fish tissue were accomplished via NEA SOP 17, Revision 3 (Appendix 23 of the QAPP).

## **SECTION 3**

### **QUALITY ASSURANCE/QUALITY CONTROL**

#### **3.1 PE SAMPLES**

GE prepared and submitted performance evaluation (PE) samples to NEA for both the 1 L and 8 L mGBM in December 2006. The PE samples contained the same 64 congeners contained in the PE samples used in the independent verification of the mGBM validation at concentrations near the current laboratory control sample (LCS) spike levels of 198 ng/L and 6 ng/L for the 1 L and 8 L mGBM, respectively. The 64 congeners are representative of those typically encountered in a Hudson River environmental sample. The laboratory summed the individual congener results on a homolog and total basis. An evaluation of the method performance was made based on acceptance limits of 70% to 130% for the homolog and total PCB results as compared to the known values. All recoveries for the homologs and total PCBs in both the 1 L and 8 L mGBM PE samples were within the 70% to 130% acceptance limits (Table 3-1).

#### **3.2 FIELD QA/QC**

QA/QC samples were collected in the field to allow evaluation of data quality. Field QA/QC samples for water column samples included equipment blank samples, blind duplicate samples, and matrix spike samples. Fish sampling does not facilitate the use of field QA/QC samples (e.g., duplicates) as part of the study design; all QA/QC samples for the fish sampling program were generated in the laboratory. The types and frequency of field QA/QC samples collected for each parameter are described below.

### **3.2.1 Field Instrument Calibration**

To ensure that field measurements completed during field data collection were collected with properly calibrated instruments, field personnel followed the manufacturer's recommendations and the procedures described below.

#### **3.2.1.1 Water Program**

For the water program, the YSI multi-parameter probe (Model 6920) was calibrated on a daily basis using known standards for turbidity, pH, and conductivity prior to each day's sampling events. The instrument's calibration was checked at the end of the day for calibration drift. In addition, prior to use, each major piece of equipment was cleaned, decontaminated, checked for damage, and repaired, if needed.

#### **3.2.1.2 Fish Program**

Balances used to weigh fish were calibrated each day prior to sampling. Calibration checks were recorded on a field log. The conductivity meter was calibrated once prior to the start of sampling each season. A Lamotte Model 2020 Portable Turbidity meter was used at each station. The turbidity meter was checked with a known turbidity solution prior to use at each station in accordance with the users manual. Field calibration activities were noted in a field log notebook. The global positioning system (GPS) on each sampling vessel had a daily check on a point with known coordinates. Equipment was maintained and repaired in accordance with manufacturer's specifications (Section B6 of the QAPP; QEA and ESI 2004). In addition, prior to use, each major piece of equipment was cleaned, decontaminated, checked for damage, and repaired, if needed.

### **3.2.2 Equipment Blanks**

Equipment blank samples were collected at the rate of 5% of the total number of environmental water samples or one per sample batch of up to 20 samples. Equipment blanks

were not required for fish tissue samples in the approved QAPP (QEA and ESI 2004). Equipment blanks for water sampling were collected using a representative clean, individual sample container used for sub-sample collection in accordance with the water column sample collection SOP (Appendix 1 of the QAPP) and CAM 001.

A volume of reagent water was obtained in the composite container equal to the Hudson River water samples to represent the entire sample collection process.

### **3.2.3 Field Duplicates**

Field duplicate samples for water were collected and submitted to the analytical laboratory “blind” without any indication of the actual sample location. Because it is impossible to collect field duplicates for fish samples, duplicates for fish were generated in the laboratory by splitting the homogenate. Duplicates were prepared at the rate of 5% of the total number of environmental samples or one per sample batch of up to 20 samples.

### **3.2.4 Laboratory Duplicates/Matrix Spikes /Matrix Spike Duplicates**

Laboratory duplicates (LDs) were typically substituted for matrix spikes (MS) or matrix spike duplicates (MSDs) for inorganic and wet chemistry analysis. Either MSDs or LDs were performed on fish samples, but not both. MS/MSDs/LDs were analyzed at the rate of one pair per sample batch (up to 20 samples) for fish samples. The water program included the analysis of MS samples at a rate of one per sample batch (up to 20 samples) and analysis of MSDs at a rate of one per month. Each MS consisted of an aliquot of laboratory-fortified environmental sample. The MS samples were extracted and analyzed following procedures used for actual sample analysis.

### **3.2.5 Hudson River Reference Material**

The BMP fish program included provisions for the analysis of Hudson River Reference Material (HRRM - a NYSDEC-developed PE sample), if available, at a rate of one per fifty

samples as a performance measure for PCB Aroclor analysis. The final HRRM, inclusive of documented acceptance limits, was not available prior to the fish monitoring program, so this QA/QC aspect of the program was not included in 2006.

### **3.3 LAB QA/QC**

#### **3.3.1 Method Blanks**

Method blanks were prepared and analyzed by the contract laboratories at a rate of at least one per analytical batch. Method blanks for water consisted of laboratory-prepared blank water processed along with the batch of environmental samples including all manipulations performed on actual samples. Method blanks for fish consisted of sodium sulfate processed, along with the batch of environmental samples, including all manipulations performed on actual samples.

#### **3.3.2 Laboratory Control Spikes**

Laboratory Control Spikes (LCSs) were analyzed at the rate of one per sample batch (up to 20 samples). LCSs consisted of laboratory-fortified method blanks. The purpose of analyzing laboratory control samples is to demonstrate the accuracy of the analytical method.

#### **3.3.3 Temperature Blanks**

A temperature blank was provided in each cooler sent from the laboratory to the field. The purpose of this sample was to document the temperature of the cooler upon arrival at the lab.

### **3.4 EPA SPLIT SAMPLES**

EPA did not collect split samples during 2006.

### **3.5 FIELD AND LABORATORY AUDITS**

A field audit of the 2006 water column collection activities performed by QEA field personnel was conducted by ESI on July 19, 2006. A second field audit of the 2006 water column collection activities was not performed in the fall of 2006 due to scheduling conflicts. A field audit of 2006 fall fish collection activities performed by QEA field personnel was conducted by ESI on August 29, 2006 (previous fish audits were on spring collection activities). The audits were conducted as described in the QAPP (Section C1.1.2.3; QEA and ESI 2004). The field audits indicated that the field crews conducted their work in a professional manner and complied with the procedures outlined in the QAPP and applicable SOPs. Additionally, the field audits indicated that consistent sample collection and processing procedures were used during 2006. A few minor issues were identified during the audits and are discussed in the audit reports (Appendix B). The issues identified in the audit reports did not jeopardize the data quality objectives of the project. When possible, the recommendations were discussed with the field team at the time of occurrence. A debriefing meeting was held with QEA field personnel at the conclusion of each audit. The field crews incorporated recommendations, as appropriate.

A laboratory audit was conducted by ESI personnel for STL Pittsburgh (providing TAL metals and hardness analysis) on October 5, 2006. An audit of NEA (providing PCB, TSS, and organic carbon analyses) was not conducted in 2006 due to scheduling conflicts; however, NEA will be audited in 2007. The audit of STL Pittsburgh was conducted as described in Section C1.2.3.3 and Appendix 40 of the approved QAPP and to provide feedback on laboratory operating issues with respect to method compliance, laboratory systems, and good laboratory practices.

The audit report for STL Pittsburgh is included in Appendix B. The audit found that the laboratory was adhering to the project specific methods and quality assurance requirements.

### **3.6 DATA MANAGEMENT QA/QC**

Data collected under the BMP are stored in an electronic database. Specialized application modules, outlined in the subsections below, were used to automate data collection, data evaluation, and data integration.

#### **3.6.1 Field Sample Data Collection System**

Field-generated data were entered into a field database via custom-designed forms developed in Microsoft® Access®. This custom application facilitated data entry and management of the collected field data for the project by capturing, managing, and maintaining field data, including electronic COC creation, sample ID creation, and bottle label creation. These forms were also developed to limit the possibility of data entry/transcription errors by including valid value pick lists for the required fields. In addition, several data fields are populated automatically to further reduce data entry/transcription errors.

#### **3.6.2 Laboratory Data Checker**

Custom computer code was written to automate checking of the electronic data deliverables (EDDs) submitted by the analytical laboratories. EDDs submitted to the data management system were automatically checked to ensure data reliability by checking them against several criteria including valid values, data types, and format. If any errors were detected on any of the levels, the file was corrected by the laboratory prior to loading into the data management system.

#### **3.6.3 Data Verification Module**

Custom computer code was written to facilitate the data evaluation process. An automated data verification module (DVM) verifies analytical data submitted by the laboratory, reviews the data against the performance specifications provided for the project, evaluates the data, produces exception reports, and loads qualified results to the project database.

The term “verification” is used to designate the criteria-based checking of the laboratory-reported QC results against the limits defined in the QAPP (QEA and ESI 2004). This comparison was used to qualify the data. The automated electronic data verification was performed on 100% of the analytical results received using the batch quality control results provided by the laboratories in the EDDs. The specific measures evaluated during verification and the associated criteria are discussed in the QAPP, Section D2, and include:

- holding times;
- accuracy (by evaluating LCS and MS/MSD recoveries);
- precision (by evaluating LD results);
- field duplicate sample precision;
- blank contamination (laboratory method blanks and field generated blanks); and
- surrogate compound recoveries.

### **3.7 DATA VALIDATION**

Electronic data verification and data validation (where necessary) were conducted after samples were collected and analyzed. The usability of the analytical data was assessed using a tiered approach. Data initially underwent an electronic data verification, which provided the first test of the quality of the results. This automated process assessed data usability by evaluating batch quality control results. The term “verification” is used because criteria-based checking of the laboratory-reported QC results against the limits defined in the QAPP (QEA and ESI 2004) is used to qualify data. Full data validation, i.e., manual qualitative and quantitative checking, was performed on 10% of all data, as well as any other analytical results that are subject to question.

Ten percent of PCB, as well as non-PCB data, were subject to manual validation. One of the first sample delivery groups (SDGs) provided for the year for each matrix (water or fish) was selected for validation in order to identify potential issues at the beginning of the project.

Subsequent SDGs were selected randomly until the annual 10% validation goal was met for each matrix and method.

Non-PCB water data validated included:

- TAL metals;
- hardness;
- TSS;
- POC; and
- DOC.

Full validation included an evaluation of documented QA/QC measures through a review of tabulated QC summary forms and raw instrument data. The validation results were also compared to the results of the electronic verification for the same set of data, which provided an indication of the accuracy of the electronic verification process. Verification and validation findings are discussed in Section 8.

### **3.8 SAMPLE ARCHIVES**

The 2006 sample extracts generated for PCB analysis as well as the homogenized fish tissue have been archived (frozen at <-10°C for extracts and <-18°C for fish tissue) and will be maintained until EPA approves this 2006 DSR. EPA will have the option of obtaining some, or all of the archived sample extracts pursuant to the RD AOC.

## **SECTION 4**

### **ROUTINE WATER SAMPLING PROGRAM RESULTS**

As described in Section 2, the BMP water sampling program consists of routine water column sampling as well as special studies. Data presented in this section are from the routine water monitoring; data generated for the Special Studies are presented in Section 6. The sample counts presented in the tables in this section vary from station to station due to the differences in the timeframe for sampling specified in the QAPP (QEA and ESI 2004), as summarized in Table 2-1. From January through March samples were collected from the stations that were free from ice. The frequency of sampling and number of stations varied due to weather conditions. There were no samples collected the weeks of February 13 and February 27, 2006. The routine water sampling program dataset is presented in the BMP database CD-ROM (Appendix C); scanned copies of the laboratory hardcopy data packages for these data are included on a DVD in Appendix D.

#### **4.1 PCBS**

The 2006 routine water monitoring included the collection and analysis of 335 samples (285 environmental plus 50 duplicates) for congener-specific PCBs by the mGBM. Sample results ranged from non-detect to 94.93 ng/L. Summary statistics for the PCB data are presented in Table 4-1. Temporal profiles of the PCB data are presented for each routine water sampling station in upstream to downstream order in Figures 4-1 through 4-9.

#### **4.2 TSS**

During 2006 routine water monitoring, a total of 335 samples (285 environmental plus 50 duplicates) were collected and analyzed for TSS using EPA Method 160.2. Sample results ranged from non-detect (<0.9 mg/L) to 194 mg/L. A temporal plot of the TSS concentrations is provided for each station in upstream to downstream order in Figures 4-1 through 4-9. Summary statistics for routine TSS samples are presented in Table 4-2.

### **4.3 POC/DOC**

During 2006 routine water monitoring, a total of 320 samples (273 environmental plus 47 duplicates) were collected and analyzed for DOC using NEA Method NE128\_03. A total of 328 samples (279 environmental plus 49 duplicates) were collected and analyzed for POC using NEA Method NE128\_03. Sample results for DOC ranged from 2.15 to 6.67 mg/L. Sample results for POC ranged from 0.15 to 4.41 mg/L. Summary statistics for DOC and POC data are presented in Table 4-3.

### **4.4 TAL METALS**

During 2006 routine water monitoring, a total of 105 samples (90 environmental plus 15 duplicates) were collected and analyzed for total and dissolved TAL metals. Total cadmium, beryllium, and silver and dissolved beryllium, chromium, and silver concentrations were below the method detection limit for all stations in 2006. Summaries of total and dissolved TAL metal results are presented in Tables 4-4 and 4-5, respectively.

### **4.5 WATER QUALITY PARAMETERS**

At each sampling location, water quality measurements were taken at mid-depth in the water column. Measurements of temperature, conductivity, pH, DO, and turbidity were taken using a YSI 6920 multi-parameter probe (Table 4-6). Prior to each day's sampling activities, the instrument is calibrated against standards to verify that the probe for each parameter is working correctly. However, once in the field, there are several factors that can influence the probe's output. These include environmental factors such as variability in air temperatures (especially in winter) between the controlled conditions under which the instrument is calibrated or transported compared to the field conditions that the probe is exposed to during deployment. Additionally, the probes can come in contact with debris during deployment. These factors may cause

degradation of membranes and other components of the instrumentation in the field during use, resulting in the collection of inaccurate data.

The data collected by the probe are downloaded and reviewed during routine QA/QC checks. In the event the data appear to have been influenced by a faulty reading in the field (such as negative readings, or values that are well outside of the range of data normally measured), the data are moved from the parameter list to the comments section of the database along with a description of why the value was qualified. The results of water quality parameter measurements are included in the project database (Appendix C).

#### **4.6 OTHER DATA COLLECTION ACTIVITIES**

Other data collection activities included obtaining daily mean flow recorded at the Fort Edward and Waterford USGS gauging stations. In addition, meteorological data was obtained from Northeast Regional Climate Center at Cornell University (NRCC 2006) for three locations near the river (Glens Falls Airport, Saratoga Springs, and Sunderland 2). The flow and meteorological data have been entered into a database (Appendix E). Other sampling related observations noted in the field are included in the project database in Appendix C.

## **SECTION 5 FISH PROGRAM RESULTS**

### **5.1 PCBS**

This section presents the results of PCB analyses performed on fish. For each species, a spatial plot of PCB concentrations is provided and summary statistics by river pool are included in tables. A total of 542 fish were collected from the Hudson River during the 2006 field sampling season (374 samples in spring, 168 samples in late summer). 542 samples were submitted for Aroclor PCB analysis using Method SW846 8082 (NE148\_04). Ten percent of the total number of fish analyzed for Aroclor PCBs (54) were also analyzed for congener-specific PCBs using Method NE013\_07. Of the 54 samples analyzed for congener-specific PCBs, 37 were collected during the spring sampling, and 17 were collected during the late summer sampling. PCBs were detected in all fish analyzed using the congener-specific analytical method. A comparison of PCB concentrations measured using Aroclor and congener-specific methods is presented in Figure 5-1. The fish sampling program dataset is presented in the BMP database CD-ROM (Appendix C); scanned copies of the laboratory hardcopy data packages for these data are included on a CD-ROM in Appendix D.

#### **5.1.1 Black Bass**

During baseline monitoring in 2006, 126 black bass (largemouth bass and smallmouth bass) were collected from the Hudson River. Aroclor PCBs were detected in 122 samples (Table 5-1, Figure 5-2). Thirteen black bass were also submitted for congener-specific PCB analysis. Congener-specific PCBs were detected in all 13 samples (Table 5-2).

#### **5.1.2 Ictalurids**

During baseline monitoring in 2006, 122 ictalurids (brown bullhead, yellow bullhead, channel catfish, and white catfish) were collected from the Hudson River. Of these, Aroclor

PCBs were detected in 118 samples (Table 5-3, Figure 5-3). Thirteen ictalurid samples were also submitted for congener-specific analysis. Congener-specific PCBs were detected in all 13 ictalurids (Table 5-4).

### **5.1.3 Perch**

During baseline monitoring in 2006, 126 perch (yellow perch and white perch) were collected from the Hudson River and submitted for Aroclor PCB analysis. Of these, Aroclor PCBs were detected in 115 samples (Table 5-5, Figure 5-4). Eleven perch were also submitted for congener-specific PCB analysis. Congener-specific PCBs were detected in all 11 samples (Table 5-6).

### **5.1.4 Pumpkinseed**

During baseline monitoring in 2006, 125 pumpkinseed were collected from the Hudson River. Aroclor PCBs were detected in all samples (Table 5-7, Figure 5-5). Seven pumpkinseed were also submitted for congener-specific PCB analysis. Congener-specific PCBs were detected in all seven samples (Table 5-8).

### **5.1.5 Forage Fish**

A total of 43 forage fish (spottail shiner, bluntnose minnow, spotfin shiner, fallfish, and mimic shiner) composites were collected from the Hudson River during the 2006 sampling season. Of these, Aroclor PCBs were detected in all samples (Table 5-9, Figure 5-6). Ten forage fish composites were also submitted for congener-specific PCB analysis. Congener-specific PCBs were detected in all 10 samples (Table 5-10).

## **5.2 LIPIDS**

Lipid results for fish are presented in this section by species. Summary statistics are included in tables for each species by river pool. A total of 542 fish were collected from the Hudson River during the 2006 field sampling season (374 samples in spring, 168 samples in fall). Percent lipid was measured on all 542 samples using Method NE158\_03. The lipid results are included in the fish dataset presented in the BMP database CD-ROM (Appendix C); scanned copies of the laboratory hardcopy data packages for these data are included on a DVD in Appendix D.

### **5.2.1 Black Bass**

During baseline monitoring in 2006, percent lipid was measured in 126 black bass (largemouth bass and smallmouth bass) fillet samples collected from the Hudson River (Table 5-11).

### **5.2.2 Ictalurids**

During baseline monitoring in 2006, percent lipid was measured in 122 ictalurid fillet samples (brown bullhead, yellow bullhead, channel catfish, and white catfish) collected from the Hudson River (Table 5-12).

### **5.2.3 Perch**

During baseline monitoring in 2006, percent lipid was measured in 126 perch (yellow perch and white perch) fillet samples collected from the Hudson River (Table 5-13).

### **5.2.4 Pumpkinseed**

During baseline monitoring in 2006, percent lipid was measured in 125 whole body pumpkinseed collected from the Hudson River (Table 5-14).

### **5.2.5 Forage Fish**

A total of 43 forage fish (common shiner, fallfish, mimic shiner, spotfin shiner, and spottail shiner) composites were collected from the Hudson River during the 2006 sampling season; percent lipid was measured in all samples (Table 5-15).

## **5.3 SEX**

Results for fish sexing are presented in this section by species. Summary statistics are included in tables for each species by river pool. A total of 542 fish were collected from the Hudson River during the 2006 field sampling season (374 samples in spring, 168 samples in fall). When it could be determined, the sex was identified for each individual collected in the spring. The fish sex results are included in the fish dataset presented in the BMP database CD-ROM (Appendix C).

### **5.3.1 Black Bass**

During baseline monitoring in 2006, fish sex was determined in 118 black bass (largemouth bass and smallmouth bass) collected from the Hudson River with 52 males and 66 females. Sex could not be determined in 8 individuals (Table 5-16).

### **5.3.2 Ictalurids**

During baseline monitoring in 2006, fish sex was determined in 122 ictalurid samples (brown bullhead, yellow bullhead, channel catfish, and white catfish) collected from the Hudson River with 62 males and 60 females (Table 5-17).

### **5.3.3 Perch**

During baseline monitoring in 2006, fish sex was determined in 109 perch (yellow perch and white perch) samples collected from the Hudson River with 79 males and 30 females. Sex could not be determined in 17 individuals (Table 5-18).

## **5.4 FIELD OBSERVATIONS**

Fish condition was assessed using field measurements and field observations. Observed external abnormalities were recorded to assess fish condition. Ictalurids appeared to present the most external abnormalities. Of the ictalurids captured from the Feeder Dam Pool, three showed signs of melanoma, one had burned barbells, and one had scoliosis. One of the largemouth bass had a wound near the dorsal fin and a smallmouth bass had black spot.

Of the ictalurids captured from Thompson Island Pool, three showed signs of melanoma, one was blind in the left eye, two had lamprey wounds, two had burned whiskers, one had a lesion on the right maxilla, one had a lesion on the ventral surface, two showed signs of fin erosion, and one had burned barbells. One largemouth bass showed signs fin erosion. Of the smallmouth bass that were captured from Thompson Island Pool, one had a left pelvic fin clip, one had damage to the left eye, one had a damaged right eye, two had black spot, and one had a lamprey attached. Of the yellow perch captured one showed signs of fin erosion and one had a wound on the left side.

Of the ictalurids captured from the Northumberland/Fort Miller Pool, five showed signs of burned barbells, three showed evidence of melanoma, ten had lesions around the mouth, one had a lamprey wound, one was blind in the left eye, one had three missing barbells, one had a tumor on the mouth, one had an eroded dorsal fin, one had a papaloma, and one had lesions throughout the body. Two yellow perch from the Northumberland/Fort Miller Pool had black spot, one had skin lesions, one had a secondary infection, and one had erosion of the caudal fin. Of the largemouth bass captured one had a mouth lesion and one had erosion in the bifurcation

of the caudal fin. For the smallmouth bass captured in the Northumberland/Fort Miller pool, five showed signs of black spot, one had a missing snout, and one had a hook wound.

Of the yellow perch captured from the Stillwater Pool, two showed signs of fin erosion and five had black spot. One of the smallmouth bass captured at Stillwater Pool had a hook in its mouth and six showed signs of black spot. Of the ictalurids captured, four had burned barbells, three had lesions on the mouth, seven showed signs of melanoma, four had eroded fins, one had a fungal infection, and one had tumors.

Of the white catfish captured at Albany/Troy, one had fin erosion and one had mouth lesions. One of the largemouth bass captured had wounds near the mouth. Of the smallmouth bass captured at Albany/Troy, one had a mouth lesion, one had a hook wound, and one had a wound on the left side.

The weight and total length of captured fish were measured to assess fish condition. Condition index was determined using the following equation:

$$\text{Condition Index}(K) = \frac{\text{Weight}(g) * 100,000}{\text{Length}(mm)^3} \quad (5-1)$$

A condition index of 1.0 indicates a fish of normal condition. A condition index greater than 1.0 indicates a fish of better than average condition.

Black bass, ictalurids, perch, and pumpkinseed captured from all five pools during the 2006 BMP had a condition index greater than 1.0 (Figures 5-7 through 5-10, respectively). Forage fish captured during the 2006 BMP had a condition index less than 1.0 at all stations (Figure 5-11). Forage fish in the Feeder Dam Pool had a condition index of 0.80. Forage fish in the Thompson Island Pool had a condition index of 0.89. Forage fish in the Northumberland/Fort Miller Pool had a condition index of 0.95. Forage fish in the Stillwater Pool had a condition index of 0.91. Forage fish in the Albany/Troy pool had a condition index of 0.68 (Figure 5-11).

## **SECTION 6 SPECIAL STUDIES AND HIGH FLOW SAMPLING RESULTS**

### **6.1 HISTORICAL STATIONS**

During the 2006 BMP, eight environmental samples were collected at the historical TID-PRW2 station and three from the Schuylerville (center channel) station. These samples were submitted for PCB, TSS, and POC/DOC analysis. At TID-PRW2, PCB concentrations were above the MDL of 9.3 ng/L in seven of the eight samples, with detectable concentrations that ranged from 17.82 to 31.7 ng/L (Table 6-1, Figure 6-1). TSS concentrations at TID-PRW2 ranged from less than 0.9 to 7.74 mg/L (Table 6-2, Figure 6-1). At the historical Schuylerville station, PCB concentrations ranged from 10.7 to 43.8 ng/L and TSS concentrations ranged from 1.6 to 7.34 mg/L (Table 6-1 and Table 6-2, Figure 6-2). A summary of POC/TOC data is presented in Table 6-3. The historical data are included in the BMP database CD-ROM (Appendix C); scanned copies of the laboratory hardcopy data packages for these data are included on a DVD in Appendix D.

### **6.2 WATERFORD HIGH FLOW**

During the 2006 BMP, high flow samples were collected during seven high flow events. Twenty-nine environmental samples were submitted for PCB, TSS, DOC, and POC. PCB and TSS data are presented for each high flow event on Figure 6-3. PCB concentrations ranged from 9.67 to 265 ng/L (Table 6-1). TSS concentrations during high flow events ranged from 6.4 to 416 mg/L (Table 6-2). A summary of POC/TOC data is presented in Table 6-3. The Waterford high flow sampling data are included in the BMP database CD-ROM (Appendix C); scanned copies of the laboratory hardcopy data packages for these data are included in a DVD in Appendix D.

### **6.3 ADDITIONAL TSS SAMPLES**

During May and June 2006, TSS samples were collected twice weekly (once during routine sampling and one additional round) at TID and Schuylerville. This resulted in the collection of eight additional samples from TID and nine additional samples from Schuylerville for TSS analysis. The TSS concentrations for these additional samples ranged from non-detect to 9.18 mg/L and 1.17 to 5.09 mg/L at TID and Schuylerville, respectively. Summary statistics for additional TSS samples are incorporated into table presented in Table 6-4. The additional TSS sampling data are included in the BMP database CD-ROM (Appendix C); scanned copies of the laboratory hardcopy data packages for these data are included in a DVD in Appendix D.

## **SECTION 7**

### **POST CONSTRUCTION REMNANT DEPOSIT MONITORING RESULTS**

Over an approximate 30-year period ending in 1977, two GE capacitor manufacturing facilities in Fort Edward and Hudson Falls, New York discharged PCBs into the Upper Hudson River (Figure 7-1). Much of the PCBs were contained in sediment deposited in the pool behind the Fort Edward Dam located at Hudson River Mile (HRM)<sup>1</sup> 194.9 (Figure 7-1). Removal of the 100-year-old dam by Niagara Mohawk Power Corporation in 1973 dropped water levels in the pool. As a result, an estimated 1.5-million cubic yards of sediment deposits (referred to as the Remnant Deposits) were left along the banks of the river up to 1.5-miles upstream of Fort Edward (NUS 1984).

GE completed the in-place containment of the Remnant Deposits during the fall of 1990 (O'Brien & Gere 1996a; JL Engineering 1992). The objectives of this containment were to control the release of PCBs from the Remnant Deposits to the Hudson River and to minimize potential human exposure to PCBs as a result of direct contact or volatilization (Consent Decree 1990). Post-construction monitoring has been conducted since 1991.

Beginning in 1991, the water column of the Hudson River has been monitored for polychlorinated biphenyls (PCBs) utilizing capillary column analytical techniques with a total PCB method detection limit (MDL) of 11 ng/L (O'Brien & Gere 1992a, 1992b). This Post Construction Remnant Deposit Monitoring Program (PCRDMP) was initiated by O'Brien & Gere in 1992, and has been performed on an annual basis since. Beginning in June of 2004, GE initiated the Baseline Monitoring Program (BMP), in accordance with the Administrative Order of Consent for the Hudson River Remedial Design and Cost Recovery for the Hudson River Dredging Project (EPA/GE 2003). The water column monitoring requirements for the PCRDMP have been included in the BMP; therefore sampling activities performed to comply with the Consent Decree (Consent Decree 1990) after June 1, 2004 are being conducted as part of the BMP.

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<sup>1</sup> For reference, the HRM system begins at the southern tip of Manhattan (the Battery) in New York City, and increases traveling upstream.

The PCRDMF consisted of water column data collection and reporting for stations located at Bakers Falls and at the Route 197 Bridge (Section 2.1, Figure 7-1). Additionally, routine water column samples were collected from a location at the base of Bakers Falls in the vicinity of the Hudson Falls Plant site on a weekly basis throughout 2006. This location, designated as BOATLAUNCH, is illustrated in Figure 7-1. This monitoring is not required by the PCRDMF Consent Decree (Consent Decree 1990) or the Consent Decree for the GE Hudson Falls plant site. These data are routinely reported to NYSDEC (Hudson Falls Plant Site Weekly Status Reports; NYSDEC site code 5-58-013, GE 2006).

The remedial action performed on the Remnant Deposits continued to be an effective measure for controlling the migration of PCBs to the Hudson River in 2006. The primary evidence for this is that the increase in PCB concentrations observed at the Route 197 Bridge compared to background conditions is minimal (typically only 2 to 3 ng/L higher than Bakers Falls; Figure 7-2). Additionally, monitoring performed in the Hudson River adjacent to the GE Hudson Falls plant site indicate that the area continued to contribute PCBs to the water column during 2006. Increased concentrations detected in the vicinity of the Hudson Falls Plant Site (relative to the background station at Bakers Falls) generally correlate with increases in PCB concentrations at Rogers Island. This condition indicates that the Boat Launch sampling station is useful as qualitative indicator of the magnitude of the GE Hudson Falls Plant Site area source.

## **SECTION 8 DATA QUALITY**

### **8.1 PE PROGRAM**

PE samples were submitted to NEA for the 1 L and 8 L mGBM as required by Section C1.2.1 of the BMP QAPP. The results of the PE sample analysis have been previously described in Section 3.2.

### **8.2 VALIDATION / VERIFICATION**

#### **8.2.1 Data Verification and Validation Results for Water Samples**

Electronic data verification and data validation were conducted, as described in Section 3.8, after samples were collected and analyzed to provide an understanding of the analytical data quality. During 2006, 10% of the environmental samples were manually validated. The number of 2006 samples validated for each method is described in Section 3.7. Additionally, Appendix F provides a listing of each 2006 sample that was validated for each method and laboratory. Appendix G provides copies of the six data validation reports prepared for each group of 2006 sample data that were validated. These reports provide the specific details of the data qualification resulting from the validation process.

Validation qualifier codes were placed next to the results in the GE analytical database so that data users can quickly assess the qualitative and/or quantitative reliability of any result. The analytical database was then used to generate tabulated reports (data tables) of the validation results and qualifier codes. The final validated results for each data set are presented as data tables in each data validation report included in Appendix G.

The same qualifier codes were used for both the data verification and validation processes. The qualifier codes and definitions used for the data were as follows:

- “Null” - No qualifier code. The compound was detected and should be considered quantitatively and qualitatively valid based on the QC reviewed.
- U - The compound/analyte was analyzed for, but was not detected above the reported sample detection limit.
- <J - The sum of the positive PCB congener peaks for the sample is greater than 0 but is below the sample-specific total PCB MDL. Quantitation is approximate (estimated).
- U\* - This compound/analyte should be considered “not detected” since it was detected in a blank at a similar level.
- J - Quantitation is approximate (estimated) due to limitations identified during the quality assurance review (data validation).
- N - The analysis indicates that there is presumptive evidence to make a “tentative identification” of this compound/analyte.
- R - Unusable (rejected) result – compound/analyte may or may not be present in this sample.
- UR - Unusable “not-detected” result; compound may or may not be present in this sample.
- UJ - This compound/analyte was not detected, but the quantitation/detection limit is probably higher than reported due to a low bias identified during the quality assurance review.

The validation qualifier code field of the GE analytical database was queried to provide a tabulation of the number of results for each analysis fraction that was valid as reported (unqualified results and non-detected results, U and <J for total PCBs only) and that was qualified with each qualifier code identified above. The percent usable and unusable data and the percent completeness were calculated for each analysis fraction according to the following equations:

$$\begin{aligned}
\% \text{ Usable Data} &= \frac{\text{Unqualified Positive Results} + \#U (+\#<J \text{ for Total PCBs}) + \#U^* + \#J + \#JN + \#UJ}{\text{Total Number of Results}} \\
\% \text{ Unusable Data} &= \frac{\#R + \#UR}{\text{Total Number of Results}} \\
\% \text{ Completeness} &= \frac{\text{Valid Data as Reported} [\text{Unqualified Positive Results} + \#U]}{[\text{Total Number of Results} - \text{positive results } <RL - <J]}
\end{aligned}$$

The percent completeness calculation does not include results qualified as estimated values (“J”) due to being below the sample-specific reporting limit but above the MDL and total PCB results qualified as <J for being above 0 but below the sample-specific MDL. These results are not included in the completeness calculation because they are estimated values pursuant to a standard EPA analytical data reporting convention.

A summary of the data quality for the individual analytical fractions is presented in the following sections. The data quality has been described based on the percent completeness and percent usable results as follows:

Qualitative Data Quality (QDQ)	% Completeness	% Usable
Excellent	95%	100%
Very Good	85%	95%
Good	75%	90%
Above Average	65%	85%
Average	45%	80%
Poor	<45%	<80%

The percent completeness goal stated in the QAPP (QEA and ESI 2004) is 95%. The above Qualitative Data Quality (QDQ) index was based on professional judgment and experience. It was developed to provide a qualitative framework to discuss the data quality. Although the description of data quality has been based on criteria for both the percent completeness and percent usable data calculations, the percent usable data calculation is a more critical reflection of the data quality than the percent completeness calculation. Percent completeness reflects the percentage of the data that satisfied all of the DQOs (i.e., the percentage of unqualified data), whereas percent usability reflects the percentage of the data that has some qualitative and/or quantitative use, which is inclusive of the data that satisfied all of the DQOs. The results of the percent completeness calculation do not indicate the nature of the

qualification of the “incomplete” data. The data which are usable but qualitatively or quantitatively qualified (i.e., the difference between the percent usable data and the percent completeness) may have no impact on the end use of the data, depending on what decisions need to be made based on that data. In other words, data that have low percent completeness may still be “100% usable” for decision-making purposes.

The following example calculations are provided based on the percent completeness, percent unusable, and percent usable data presented on Table 8-1 for PCB congeners (whole water extraction) (NE207\_03) and following the explanations in Notes 6, 7, and 8:

1. Percent Completeness is the sum of results that were valid as reported [Unqualified Positive Results + U]/[Total Number of Results - J<sup>4</sup> - <J].

$$\text{Ex. } 94.8\% = [(5,127 + 31,198)/(44,183 - 5,809 - 65)]*100$$

2. Percent Unusable Data is the sum of the results qualified R + UR/Total Number of Results.

$$\text{Ex. } 0.17\% = [(0 + 76)/44,183]*100$$

3. Percent Usable Data is the sum of the Unqualified Positive Results + U [+<J for Total PCBs] + U\* + J + JN + UJ/Total Number of Results.

$$\text{Ex. } 99.8\% = [(5,127 + 31,198 + 65 + 1,519 + 6,070 + 0 + 128)/44,183]*100$$

The overall data quality for the water sample data is very good and the vast majority of the results are usable (Table 8-1). The percent usable data, percent unusable data, and percent completeness for the entire water data set are 99.8%, 0.16%, and 92.5%, respectively. The overall data quality for the fish tissue sample data is excellent and all of the results are usable (Table 8-2). The percent usable data, percent unusable data, and percent completeness for the entire fish tissue data set are 100.0%, 0.0%, and 95.2%, respectively.

### ***8.2.1.1 Data Verification and Validation Results for PCBs Congeners***

The data quality for the water samples for PCB congeners (whole water extraction) analyzed by NE207\_03 is very good (Table 8-1). The percent usable data, percent unusable data, and percent completeness for the entire PCB congeners (whole water extraction) data set are 99.8%, 0.17%, and 94.6%, respectively.

The data verification module used to verify the PCB analysis data tracks the reason(s) that sample results are qualified for the individual assessment measures (e.g., holding times). The GE database was queried to determine why those data were qualified, but results from manual validation are not tracked in the GE analytical database. Thus, the validation reports were also evaluated manually. This combined assessment indicated that the electronic data verification process identified the primary quality control measures that resulted in qualification of data, as listed below in order of decreasing frequency:

- Blank contamination – Positive sample results that exhibited PCB concentrations similar to that in the field and method blanks were qualified as “not-detected” and flagged “U\*.” Qualification due to blank contamination occurred for approximately 3.4% of the PCB congener (whole water extraction) data set and was limited to individual PCB congener results.
- Total PCB results summed from estimated individual congener results – The Total PCB results in all samples (0.88% of results) were qualified as estimated because at least one of the individual congener results that were summed to calculate the Total PCB result was qualified as estimated.
- MS or MSD recoveries outside of acceptance criteria – Water sample results associated with MS recoveries outside of acceptance criteria (outside of 60-140%) resulted in qualification of “not-detected” results as unusable “UR” for approximately 0.17% and positive and “not-detected” results as estimated “J” and “UJ”, respectively for approximately an additional 0.59% of the PCB congener (whole water extraction) data set.

- Field duplicate precision – Water sample results associated with original and field duplicate samples that did not meet the project laboratory replicate precision criteria resulted in qualification of positive and “not-detected” results as estimated “J” and “UJ”, respectively for approximately 0.14% of the PCB congener (whole water extraction) data set.

As the above list indicates, qualification of data occurred primarily from blank contamination and MS/MSD recoveries that were outside of criteria. Additionally, approximately 13% of the data were qualified as estimated “J” due to the standard EPA analytical data reporting convention of qualifying data as estimated when they fall between the reporting limit and the MDL.

#### ***8.2.1.2 Data Verification and Validation Results for Other Parameters***

The data quality for total metals and dissolved metals by EPA Method 200.8 is good and above average, respectively (Table 8-1). The percent usable data, percent unusable data, and percent completeness for the total metals by EPA Method 200.8 data set are 100%, 0.0%, and 79.9%, respectively. The percent usable data, percent unusable data, and percent completeness for the dissolved metals by EPA Method 200.8 data set are 99.9%, 0.10%, and 70.0%, respectively. The queries of the GE database and manual evaluation of the data validation reports revealed that metals sample results were qualified for the following reasons, listed in order of decreasing frequency:

- Blank contamination – Qualification as “U\*”, due to field, method, or calibration blank contamination occurred for 20% of the total and dissolved metals sample results (16% of the total metals results and 24% of the dissolved metals results).
- Field duplicate precision – Water sample results associated with original and field duplicate samples that did not meet the project field duplicate precision criteria resulted in qualification of positive and “not-detected” results as estimated “J” and “UJ”, respectively for approximately 0.33% of the samples results (0.65% of the dissolved metals results).

- Laboratory duplicate precision – Water sample results associated with original and field duplicate samples that did not meet the project field duplicate precision criteria resulted in qualification of positive and “not-detected” results as estimated “J” and “UJ”, respectively for approximately 0.30% of the samples results (0.60% of the dissolved metals results).
- Negative calibration verification blanks – Water sample results associated with calibration verification blanks with negative results with absolute values greater than two-times the method detection limit (MDL) resulted in qualification of “not-detected” results for one analyte as estimated “UJ” for 1 SDG (0.33% of the sample results).
- Dissolved metal results significantly greater than total metal results – Water sample results where the dissolved metal result was significantly greater than the total metal result resulted in qualification of positive results as estimated “J” for approximately 0.20% of the metal sample results.
- Serial dilution precision – Water sample results associated with a serial dilution outside of precision criteria results in qualification of positive results for 1 total metal as estimated “J” for 1 SDG (0.15% of the sample results).
- Matrix spike (MS) recoveries outside of acceptance criteria – Water sample results associated with MS recoveries outside of acceptance criteria resulted in qualification of “not-detected” results as unusable “UR” for approximately 0.05% of the metals sample results (0.10% of the dissolved metals results) and positive results as estimated “J” for approximately 0.05% of the metals sample results (0.10% of the dissolved metals results).

Qualification of total and dissolved metals by EPA 200.8 data occurred primarily due to the blank contamination. Additionally, approximately 17% of the total and dissolved metals by EPA 200.8 data were qualified as estimated “J” pursuant to the standard EPA analytical data reporting convention of qualifying data as estimated that fall between the reporting limit and the MDL.

The data quality for total and dissolved mercury is very good (Table 8-1). The percent usable data, percent unusable data, and percent completeness for the total mercury data set are

100.0%, 0.0%, and 94.0%, respectively. The percent usable data, percent unusable data, and percent completeness for the dissolved mercury data set are 100.0%, 0.0%, and 92.0%, respectively. The queries of the GE database revealed that five total mercury sample results and seven dissolved mercury sample results were qualified due to blank contamination (6.7% of the mercury data). Approximately 5.0% of the mercury sample results were qualified as “J” pursuant to the standard EPA analytical data reporting convention of qualifying data as estimated that fall between the reporting limit and the MDL.

The data quality for hardness by EPA 130.2 is excellent (Table 8-1). The percent usable data, percent unusable data, and percent completeness for the hardness data set are 100.0%, 0.0%, and 97.8%, respectively. The queries of the GE database and manual evaluation of the data validation reports revealed that two hardness sample results (2.2% of the hardness data) were qualified for field duplicate imprecision.

The data quality for TSS by EPA 160.2 is excellent (Table 8-1). The percent usable data, percent unusable data, and percent completeness for the TSS data set are 100.0%, 0.0%, and 94.8%, respectively. The queries of the GE database and manual evaluation of the data validation reports revealed that TSS sample results were qualified for the following reasons, listed in order of decreasing frequency:

- Field duplicate precision – Qualification of positive results as estimated “J” due to field duplicate imprecision occurred for approximately 1.9% of the TSS sample results.
- Laboratory replicate precision – Water sample results associated with original and laboratory replicate samples that did not meet the project laboratory replicate precision criteria resulted in qualification of positive results as estimated “J” for approximately 1.3% of the TSS sample results.
- Laboratory control sample recoveries outside of acceptance criteria – Water sample results associated with LCS recoveries outside of acceptance criteria resulted in qualification of positive results as estimated “J” for approximately 1.3% of the sample results.

- Holding time – Positive results were qualified as estimated “J”, when analysis holding times were exceeded. Qualification due to exceedance of the analysis holding time occurred in four samples or approximately 1.1% of the TSS sample results.

All of the TSS data are usable, but approximately 5.2% were qualified as estimated “J” or “UJ”, due to the issues listed above. Qualification of TSS data occurred primarily due to field duplicate and laboratory replicate imprecision, LCS recoveries outside of criteria, and exceeded holding times.

The data quality for POC/DTC/DOC is average (Table 8-1). The percent usable data, percent unusable data, and percent completeness for the POC/DOC data set are 100.0%, 0.0%, and 46.1%, respectively. The queries of the GE database and manual evaluation of the data validation reports revealed that POC/DTC/DOC sample results were qualified for the following reasons, listed in order of decreasing frequency:

- Holding time – Positive results were qualified as estimated “J”, when analysis holding times were exceeded. Qualification due to exceedance of the analysis holding time occurred in approximately 32% of the POC/DTC/DOC sample results.
- Blank contamination – Qualification as “U\*”, due to method or field blank contamination occurred for 31% of the POC/DTC/DOC sample results.
- Field duplicate precision – Qualification of positive results as estimated “J”, due to field duplicate imprecision occurred for approximately 8.8% of the POC/DTC/DOC sample results.

All the POC/DTC/DOC data are usable, but approximately 22% were qualified as estimated “J” or “UJ”, 34% were qualified due to blank contamination due to the issues listed above. Qualification of POC/DTC/DOC data occurred primarily due to blank contamination and exceeded holding times.

## 8.2.2 Data Verification and Validation Results for Fish Tissue Samples

### 8.2.2.1 Data Verification and Validation Results for PCBs as Aroclors

The data quality for PCBs as Aroclors in fish tissue analyzed by method NE148\_04 is excellent (Table 8-2). The percent usable data, percent unusable data, and percent completeness for the entire PCBs as Aroclors data set are 100.0%, 0.00%, and 95.8%, respectively. None of the data was qualified as unusable.

The data verification module used to verify the PCB analysis data tracks the reason(s) that sample results are qualified for the individual assessment measures (i.e., holding times). The GE database was queried to determine why those data were qualified, but results from manual validation are not tracked in the GE analytical database. Thus, the validation reports were also evaluated manually. This combined assessment indicated that the electronic data verification process identifies the primary quality control measures that resulted in qualification of data, as listed below in order of decreasing frequency:

- Insufficient extraction time - All samples in one SDG were extracted for 1 hour less than the minimum extraction duration of 16 hours, which resulted in qualification of positive and “not-detected” results as estimated “J” and “UJ”, respectively for approximately 4.6% of the samples results.
- Laboratory replicate precision – Fish tissue sample results associated with original and laboratory replicate samples that did not meet the project laboratory replicate precision criteria resulted in qualification of positive results as estimated “J” for approximately 0.30% of the samples results.

As the above list indicates, qualification of data as estimated “J” or “UJ” occurred primarily from the insufficient extraction time and laboratory replicate imprecision. Additionally, approximately 0.35% of the data were qualified as estimated “J” due to the standard EPA analytical data reporting convention of qualifying data as estimated when they fall between the reporting limit and the MDL.

#### ***8.2.2.2 Data Verification and Validation Results for PCB Congeners***

The data quality for the fish tissue sample PCBs congeners analyzed by NE013\_07 is excellent (Table 8-2). The percent usable data, percent unusable data, and percent completeness for the entire PCBs as Aroclors data set are 100.0%, 0.0%, and 94.7%, respectively. None of the data was qualified as unusable. The queries of the GE database revealed that the PCB congener sample results were qualified for the following reasons, listed in order of decreasing frequency:

- Blank contamination – Positive sample results that exhibited PCB concentrations similar to that in the method blanks were qualified as “not-detected” and flagged “U\*”. Qualification due to blank contamination occurred for approximately 3.3% of the sample results and was limited to individual PCB congener results.
- Total PCB results summed from estimated individual congener results – The Total PCB results in all samples (0.89% of results) were qualified as estimated because at least one of the individual congener results that were summed to calculate the Total PCB result was qualified as estimated.
- Laboratory replicate precision – Fish tissue sample results associated with original and laboratory replicate samples that did not meet the project laboratory replicate precision criteria resulted in qualification of one positive result as estimated “J” for approximately 0.016% of the samples results.

As the above list indicates, qualification of data occurred primarily from blank contamination. Additionally, approximately 26% of the data were qualified as estimated “J” due to the standard EPA analytical data reporting convention of qualifying data as estimated when they fall between the reporting limit and the MDL.

### 8.3 FIELD DUPLICATES

Water field duplicates were submitted for analysis by NE207\_03 (PCB congeners), EPA 200.8 (total and dissolved ICP/MS metals), EPA 245.1 (total and dissolved mercury), EPA 130.2 (hardness), EPA 160.2 (TSS), and NE128\_03 (POC, DTC, DOC). Field duplicates were prepared in the field at the rate of 5% of the total number of environmental samples or one per sample batch of up to 20 samples. Fish tissue field duplicates were not submitted for analysis because it is impossible to collect field duplicates for fish samples.

The precision criteria for field duplicate pairs are presented in Section B5.1.2 of the QAPP (QEA and ESI 2004). For field duplicate pairs where both results were greater than or equal to five times the reporting limit, the precision criterion is that the relative percent difference (RPD) between the results should be less than or equal to 35% for PCB congeners and less than or equal to 20% for all other parameters. For field duplicate pairs where at least one of the results was less than five times the reporting limit (including when one result was a non-detect), the precision criterion is that the difference between the results should be less than or equal to the reporting limit. A value of half the reporting limit was used for not-detected results in the difference calculation. If the analyte is not detected in the sample or the field duplicate sample, the RPD is not calculated and a quantitative evaluation is not made since neither sample had a positive result.

#### 8.3.1 Field Duplicate Results for PCBs

A summary of the field duplicate results for samples analyzed by the mGBM (NE207\_03) is presented in Table 8-3. The table includes the following information:

- The total number of field duplicate pairs is presented in the column with the heading “Total No. Field Duplicate Pairs”. The table presents the total number of field duplicate pairs for each analyte as well as the total number of field duplicate result pairs.
- The total number of the field duplicate pairs that had not-detected results in both the parent sample and field duplicate is presented in the column with the heading “Total No.

Field Duplicate Pairs with NDs for Both Samples” (All of these meet field duplicate precision criteria because both results are “not-detected”). This information is also presented by analyte.

- The total number of the field duplicate pairs that had positive results in the field duplicate and/or parent sample is presented in the columns under the heading “Total No. Field Duplicate Pairs with Positives in Either Sample”. The total number (“Total No.”), the number that met criteria (“No. Meet Criteria”) and that did not meet criteria (“No. Do Not Meet Criteria”), and the percentage that met criteria (“% Meet Criteria”) and did not meet criteria (“% Do Not Meet Criteria”) are presented. This information is also presented by analyte.
- The overall percentage of results that met criteria is presented in the column with the heading “Overall % Meet Criteria”. This information is also presented by analyte.

A total of 53 field duplicate pairs were analyzed for PCB congeners by the mGBM (NE207\_03); a very high percentage (99%) of the results met the field duplicate precision criteria. For Total PCBs, all of the results met the field duplicate precision criteria. For the individual PCB congeners, the percentage of results that met the field duplicate precision criteria ranged from 83% to 100%. The percentage of field duplicate pairs with positive results in either sample that met the field duplicate precision criteria was high for all analytes (95%) and for Total PCBs (100%).

### **8.3.2 Field Duplicate Results for Other Parameters**

A summary of the field duplicate results for samples analyzed by methods 200.8, 245.1, 130.2, 160.2, and NE128\_03, is presented in Table 8-4. The table includes the following information:

- For each method, the total number of field duplicate pairs is presented in the in the column with the heading “Total No. Field Duplicate Pairs”. The table presents the total number of field duplicate pairs for each analyte as well as the total number of field duplicate result pairs.

- For each method, the total number of the field duplicate pairs that had not-detected results in both the parent sample and field duplicate is presented in the column with the heading “Total No. Field Duplicate Pairs with NDs for Both Samples” (All these meet field duplicate precision criteria because both results are “not-detected”). This information is also presented by analyte.
- For each method, the total number of the field duplicate pairs that had positive results in the field duplicate and/or parent sample is presented in the columns under the heading “Total No. Field Duplicate Pairs with Positives in Either Sample”. The total number (“Total No.”), the number that met criteria (“No. Meet Criteria”) and that did not meet criteria (“No. Do Not Meet Criteria”), and the percentage that met criteria (“% Meet Criteria”) and did not meet criteria (“% Do Not Meet Criteria”) are presented. This information is also presented by analyte.
- For each method, the overall percentage of results that met criteria is presented in the column with the heading “Overall % Meet Criteria”. This information is also presented by analyte.

Very good precision was also demonstrated by the field duplicate pair results for total and dissolved metals. A total of 15 field duplicate pairs were analyzed by methods 200.8 and 245.1. The percentages of field duplicate results that met criteria for total and dissolved metals by 200.8 are 98% and 96%, respectively. All 15 field duplicate pairs met criteria for total and dissolved mercury. Total mercury was only detected in one field duplicate pair and dissolved mercury was not detected in any field duplicate pair.

Good precision was demonstrated by the field duplicate pair results for hardness. A total of 15 field duplicate pairs were analyzed for hardness by EPA 130.2 and 87% of the results met the field duplicate precision criteria (Table 8-4).

Good precision was demonstrated by the field duplicate pair results for POC, DTC, DOC, and TSS (Table 8-4). A total of 49 field duplicate pairs were analyzed for POC and for DTC and/or DOC and 94%, 92%, and 96% respectively, of the results met the field duplicate precision

criteria. A total of 53 field duplicate pairs were analyzed for TSS and 83% of the results met field duplicate precision criteria.

#### **8.4 EQUIPMENT BLANKS**

Equipment blanks were collected to monitor external contamination during sample collection at the frequency described in Section 3.3.2. As previously indicated, equipment blanks were not collected for fish tissue samples. Summary statistics for the equipment blanks with analyte positive results greater than the MDL (other than individual PCB congener results) are presented in Table 8-5. Of the 50 equipment blanks collected for PCB analysis by the mGBM (NE207\_03), none had detectable Total PCB concentrations above the MDL (trace concentration level PCB congeners were detected in equipment blanks). In addition, positive results were not observed in any of the 15 equipment blanks collected for hardness. In general, trace concentrations of remaining analytes were detected in the equipment blanks associated with the water sampling program. Trace concentration levels were detected for the equipment blank total and dissolved metals analysis with calcium (total and dissolved), chromium (total and dissolved), copper (total and dissolved), sodium (total and dissolved), and zinc (total and dissolved) being detected in 50% or more of the blanks collected. The impacts of the equipment blank concentrations were assessed during the electronic data verification and manual data validation processes and affected sample results qualified as “U\*”.

## **SECTION 9 SUMMARY**

The objective of the BMP is to provide data to establish pre-dredging conditions where necessary for use in evaluating achievement of performance standards and provide data on PCB levels in fish and water to allow the evaluation of changes and system recovery trends. The BMP entails the routine collection and analysis of water and fish samples, as well as the performance of several special studies to support the remedial design. Data collected during the multi-year monitoring program will be used to satisfy the DQOs established in the QAPP (QEA and ESI 2004).

The routine water sampling program was continued during 2006. Weekly routine monitoring at the six Upper Hudson River stations produced a total of 308 samples for PCBs and TSS (environmental samples and duplicates) for use in establishing monthly loads and variability for performance standards monitoring. In addition, samples for POC and DOC were collected weekly, and TAL metals samples were collected biweekly. Monitoring at the Mohawk River at Cohoes, Albany, and Poughkeepsie was performed monthly to collect samples for PCB, TSS, POC, and DOC analyses. Water quality parameter data (i.e., turbidity, DO, pH, conductivity, and temperature) were collected at all stations during each sampling event. PCB, TSS, POC, and DOC samples were collected at Waterford during seven high flow events in 2006.

Several special studies were completed in 2005, and therefore were not conducted in 2006. However, PCB and TSS data were collected monthly at the historical stations at TID-PRW2 and Schuylerville (center channel). An analysis was performed to assess the correlation between the two historical stations and the BMP locations. A CAM was submitted to EPA recommending the discontinuation of these stations. Schuylerville center was discontinued in August 2006 whereas the sampling at TID-PRW2 has continued pending input from EPA. An additional sample for TSS analysis was collected from both historical stations in May and June 2006.

The BMP fish program continued in 2006 in accordance with the QAPP (QEA and ESI 2004). Adult fish were sampled in the spring and yearling pumpkinseed and forage fish were sampled in late summer. During the spring sampling event, 374 adult species of black bass (largemouth and smallmouth bass), perch (yellow or white perch), and ictalurids (brown/yellow bullhead and channel/white catfish) were collected from 15 stations in the Upper Hudson River and one location in the Lower Hudson River (below the Federal Dam in Troy). During the late summer sampling event, a total of 168 yearling pumpkinseed and forage fish were collected from the stations sampled in the spring. The forage fish were then composited into 43 samples for analyses (ten composites per pool except for Albany/Troy). A total of 542 samples (spring and late summer) were submitted for Aroclor PCB and lipid analysis. Ten percent of the total number of fish analyzed for Aroclor PCBs were also analyzed for congener-specific PCBs. Field measurements and observations were recorded for fish collected to assess overall fish condition in each pool.

## SECTION 10 REFERENCES

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## TABLES

**Table 2-1. Hudson River water monitoring summary.**

Station	Hudson RM	Sample Type <sup>1</sup>	Analyte and Sampling Frequency		
			PCBs, TSS, Suspended OC, Dissolved OC	Additional TSS	TAL Metals
Bakers Falls	197	Centroid (~center channel)	Year-round/weekly		May-Nov./bi-weekly
Rogers Island	194.2	Centroid (~center of East and West channels)	Year-round/weekly		May-Nov./bi-weekly
Thompson Island <sup>2</sup>	187.5	Transect (6 loc.)	March-Nov./weekly	Weekly (May-June)	May-Nov./bi-weekly
Schuylerville <sup>2</sup>	181.4	Transect (6 loc.)	Year-round/weekly	Weekly (May-June)	May-Nov./bi-weekly
Stillwater	168.4	Transect (5 loc.)	May-Nov./weekly		May-Nov./bi-weekly
Waterford	156	Transect (5 loc.)	Year-round/weekly		May-Nov./bi-weekly
		Centroid (~center channel)	During high flow		
Mohawk River at Cohoes	NA	Transect (5 loc.)	Year-round/monthly		
Albany/ Troy <sup>3</sup>	145	Centroid (~center channel)	May-Nov./monthly		
Poughkeepsie <sup>3</sup>	75	Centroid (~center channel)	May-Nov./monthly		

*Notes:*

*Water Quality (WQ) measurements that include temperature, specific conductivity, pH, turbidity and dissolved oxygen were taken for each water sample using a probe.*

<sup>1</sup> *A single composite sample was generated for each station.*

<sup>2</sup> *The historical single point sampling locations at TID (TID-PRW2) and Schuylerville were sampled simultaneously with the transect sampling once per month. The Schuylerville station was discontinued in August 2006.*

<sup>3</sup> *Only PCB and TSS were measured at the Lower Hudson stations.*

**Table 2-2. Fish BMP sampling locations and number of each species per location - spring 2006.**

Location		Size (TL)	SMB/LMB	BB/YB	YP/WP	Total	Sample Date	Notes	Previous Transects Sampled (2004, 05)
			>305 mm	>200 mm	>170mm/>160 mm				
		Site Code	Number of Adult Fish						
	Feeder Dam	FD1	20	20	20	60	6/4/06; 6/5/06	Transects 64, 65, 66, 67, 69, 70, 73, 74, 77, 78; plus 2119 seconds outside transects.	64, 65, 66, 73, 77
Feeder Dam Total			20	20	20	60			
Upstream	Thompson Island Pool	TD1	5	5	5	15	5/30/2006	Near Rogers Island; Transects 37, 40, 41, 45.	37, 40, 42, 43, 45
	Thompson Island Pool	TD2	5	5	5	15	5/30/2006	Near RM 193; 1927 shocking seconds.	
	Thompson Island Pool	TD3	5	5	5	15	5/31/2006	Just upstream of Snook Kill - behind islands on eastern shore; Transect 63.	63
	Thompson Island Pool	TD4	5	5	5	15	5/31/2006	Northern end of Griffin Island; Transect 54.	54
Downstream	Thompson Island Pool*	TD5	10	10	10	30	5/31/2006	Behind Griffin Island; Transects 46, 47, 48.	46, 47, 48, 49
TIP Totals			30	30	30	90			
Upstream	Ft.Miller/Northumberland Pools (LL section)	ND1	5	5	5	15	6/8/2006	From Thompson Island to small island below (around island).	
	Ft.Miller/Northumberland Pools (LL section)	ND2	5	2	6	13	6/8/2006	Short 3 bullhead; extra perch submitted.	
	Ft.Miller/Northumberland Pools	ND3	5	5	5	15	6/6/2006	Below Fort Miller dam to two small islands; including cove on east shore; 2700 shocking seconds.	
	Ft.Miller/Northumberland Pools	ND4	0	0	0	0	Site abandoned 2004 - no habitat.		
Downstream	Ft.Miller/Northumberland Pools	ND5	10	10	10	30	6/6/2006	Wetland area above Northumberland Dam; 2500 shocking seconds.	
FM/ND Totals			25	22	26	73			
Upstream	Stillwater Pool	SW1	5	5	5	15	6/1/2006	Below Battenkill; transects 18, 19, 20, 21, 22, 23, 55, 56, 57, 58, 62.	20, 21, 22, 23, 56, 57, 62
	Stillwater Pool	SW2	5	5	5	15	6/2/2006	Approx. 3/4 mile upstream of Coveville; Transects 28, 29.	28, 29, 29A
	Stillwater Pool**	SW3	10	10	10	30	6/2/2006	Coveville; transects 24, 35, 36.	24, 25, 26, 35, 36
	Stillwater Pool	SW4	5	5	5	15	6/6/2006	Near RM 173; transects 31, 32, 33.	31, 32, 33
Downstream	Stillwater Pool	SW5	5	5	5	15	6/3/2006	Just above Stillwater Dam; ~4000 shocking seconds.	
SW Totals			30	30	30	90			
	Albany/Troy	AT1	21	20	20	61	6/7/2006	18 white catfish; 2 channel catfish; 8300 shocking seconds.	
Albany/Troy Totals			21	20	20	61		Below dam to Green Island Bridge.	

Notes:

\*Historical DEC location behind Griffin Island.

\*\*Historical DEC location near Coveville.

SMB/LMB - equal numbers from each location when possible.

YP/WP equal numbers of each at Albany/Troy (10 of each).

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**Table 2-3. Fish BMP sampling locations and number of each species per location (2006 fall sampling).**

		Size (TL)	PS	STS <sup>1</sup>	Total	Sample Date	Shocking Seconds	Site Description	Notes	
			70-150 mm							
Location		Site Code	Number of Fish <sup>2</sup>							
	Feeder Dam	FD1	20	10	30	28-Aug-06	3545	Feeder Dam pool near boat launch.		
Feeder Dam Total					30					
upstream	Thompson Island Pool	TD1	5	2	7	28-Aug-06	3238 (includes TD1)	Near Rogers Island.		
	Thompson Island Pool	TD2	5	2	7	28-Aug-06		Near RM 193.		
	Thompson Island Pool	TD3	5	2	7	28-Aug-06		914	Just upstream of Snook Kill - behind three sisters islands on eastern shore.	
	Thompson Island Pool	TD4	5	2	7	28-Aug-06		2290	Northern end of Griffin Island.	
downstream	Thompson Island Pool*	TD5	10	2	12	28-Aug-06	2093	Near RM 190 - along eastern shoreline.		
TIP Totals			30	10	40					
upstream	Ft.Miller/Northumberland Pools (LL section)	ND1	0	0	0	Not sampled		From Thompson Island to small island below.	Access not available in landlocked section.	
	Ft.Miller/Northumberland Pools (LL section)	ND2	0	0	0	Not sampled		Downstream end of pool.	Access not available in landlocked section.	
	Ft.Miller/Northumberland Pools	ND3	10	5	15	29-Aug-06	3759	Below Fort Miller Dam to two small islands.	Sample size increased to account for no samples in ND1 and ND2.	
	Ft.Miller/Northumberland Pools	ND4			0			Abandoned.		
downstream	Ft.Miller/Northumberland Pools	ND5	15	5	20	29-Aug-06	1227	Wetland area above Northumberland Dam.	Sample size increased to account for no samples in ND1 and ND2.	
FM/ND Totals			25	10	35					
upstream	Stillwater Pool	SW1	5	2	7	29-Aug-06	3334	Below Battenkill.		
	Stillwater Pool	SW2	5	2	7	29-Aug-06	1790	Approx. 3/4 mile upstream of Coveville.		
	Stillwater Pool	SW3	5	2	7	29-Aug-06	2226	Coveville.		
	Stillwater Pool	SW4	5	2	7	29-Aug-06	1233	Near RM 173.		
downstream	Stillwater Pool**	SW5	10	2	12	29-Aug-06	1228	Just above Stillwater Dam.		
SW Totals			30	10	40					
	Albany/Troy	AT1	20	3	23					
Albany/Troy Totals			20	3	23	30-Aug-06	14410	Near RM 144; Albany South Turning Basin.	Very few minnows.	

<sup>1</sup> Substitute species for spottail shiner include: banded killifish, bluegill, blacknose dace, common shiner, fallfish, golden shiner, longnose dace, or tessellated darter.

<sup>2</sup> Number of composite samples for forage fish.

\*Historical DEC location across from Griffin Island (east channel).

\*\*Historical DEC location near Stillwater Dam.

**Table 3-1 - Summary of Green Bay Congener Method PE Homolog and Total PE Results**

<b>Homolog Group</b>	<b>PE</b>	<b>PE Concentration ng/L</b>	<b>Lower Control Limit (70%R) ng/L</b>	<b>Upper Control Limit (130%R) ng/L</b>
Monochlorobiphenyl	8-L	0.240	0.168	0.312
Dichlorobiphenyl	8-L	0.960	0.672	1.248
Trichlorobiphenyl	8-L	1.800	1.260	2.340
Tetrachlorobiphenyl	8-L	2.640	1.848	3.432
Pentachlorobiphenyl	8-L	1.440	1.008	1.872
Hexachlorobiphenyl	8-L	0.720	0.504	0.936
Total PCB	8-L	7.800	5.460	10.140
Monochlorobiphenyl	1-L	6.060	4.242	7.878
Dichlorobiphenyl	1-L	24.160	16.912	31.408
Trichlorobiphenyl	1-L	45.300	31.710	58.890
Tetrachlorobiphenyl	1-L	66.440	46.508	86.372
Pentachlorobiphenyl	1-L	36.240	25.368	47.112
Hexachlorobiphenyl	1-L	18.120	12.684	23.556
Total PCB	1-L	196.320	137.424	255.216

<b>Weight %</b>	<b>Conc. ng/L</b>	<b>% Recovery</b>
2.55%	0.201	83.9
11.16%	0.882	91.8
28.79%	2.275	126.4
28.37%	2.241	84.9
21.71%	1.715	119.1
7.42%	0.586	81.4
	7.772	99.6
2.38%	4.328	71.4
10.30%	18.730	77.5
28.01%	50.934	112.4
28.90%	52.552	79.1
22.53%	40.969	113.0
7.87%	14.311	79.0
	178.30	90.8

**Table 4-1. Baseline water program PCB summary statistics.**

Location	Sample Counts		Frequency Detected (%)	PCBs (ng/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
Bakers Falls	49	4	23	1.15	1.46	1.89	0.07
Rogers Island	46	6	96	1.07	2.39	9.84	0.21
Thompson Island Dam	35	6	100	9.63	34.09	94.93	2.60
Schuylerville (Transect)	50	14	91	10.07	34.51	77.64	2.25
Stillwater	31	4	100	17.57	41.46	81.64	2.57
Waterford	50	13	92	10.02	26.73	79.22	1.76
Mohawk River at Cohoes	10	2	8	13.22	13.22	13.22	--
LHR Albany	7	1	100	11.93	18.42	25.86	1.73
LHR Poughkeepsie	7	0	86	13.32	18.28	26.30	1.90

*Note:*

*Statistics based on detectable concentrations only.*

**Table 4-2. Baseline water program TSS summary statistics.**

Location	Sample Counts		Frequency Detected (%)	TSS (mg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
Bakers Falls	49	4	74	0.90	2.46	12.60	0.31
Rogers Island	46	6	83	1.00	2.70	12.30	0.37
Thompson Island Dam	35	6	83	1.12	3.70	8.10	0.32
Schuylerville (Transect)	50	14	84	1.11	6.64	90.40	1.77
Stillwater	31	4	89	1.19	7.24	54.90	1.72
Waterford	50	13	100	1.12	15.27	156.00	3.54
Mohawk River at Cohoes	10	2	100	3.71	32.80	194.00	15.01
LHR Albany	7	1	100	2.73	12.07	22.10	2.48
LHR Poughkeepsie	7	0	100	13.30	17.34	21.60	1.05

*Note:*

*Statistics based on detectable concentrations only.*

**Table 4-3. Baseline water program POC/DOC summary statistics.**

Location	Sample Counts		Frequency Detected (%)	Organic Carbon (mg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
Dissolved Total Organic Carbon							
Bakers Falls	47	4	98	3.14	4.56	6.66	0.13
Rogers Island	46	6	94	3.19	4.52	6.67	0.12
Thompson Island Dam	35	6	98	3.14	4.52	6.27	0.13
Schuylerville (Transect)	48	13	97	2.95	4.19	5.80	0.09
Stillwater	31	4	97	2.76	4.39	5.79	0.12
Waterford	48	12	98	2.77	3.90	5.45	0.09
Mohawk River at Cohoes	10	2	100	2.15	3.76	5.60	0.28
LHR Albany	4	0	100	3.31	4.19	5.18	0.40
LHR Poughkeepsie	4	0	100	2.37	3.94	4.66	0.53
Particulate Organic Carbon							
Bakers Falls	49	4	25	0.15	0.75	2.09	0.13
Rogers Island	46	6	35	0.21	0.56	0.95	0.04
Thompson Island Dam	35	6	24	0.50	0.63	0.84	0.04
Schuylerville (Transect)	50	14	31	0.20	0.63	1.74	0.08
Stillwater	31	4	20	0.41	0.81	1.61	0.15
Waterford	50	13	35	0.27	0.88	2.34	0.11
Mohawk River at Cohoes	10	2	50	0.72	1.50	4.41	0.59
LHR Albany	4	0	25	1.07	1.07	1.07	--
LHR Poughkeepsie	4	0	50	0.79	0.99	1.19	0.20

*Note:*

*Statistics based on detectable concentrations only.*

**Table 4-4. Baseline water program total TAL metals summary statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Aluminum							
Bakers Falls	15	2	94	38.50	70.15	121.00	7.21
Rogers Island	15	1	94	39.10	86.58	177.00	10.75
Thompson Island Dam	15	3	94	38.20	99.35	208.00	14.02
Schuylerville (Transect)	15	4	95	38.90	121.06	242.00	14.14
Stillwater	15	2	94	42.40	147.26	499.00	29.43
Waterford	15	3	100	8.00	182.14	619.00	36.77
TAL - Antimony							
Bakers Falls	15	2	12	0.06	0.08	0.10	0.02
Rogers Island	15	1	19	0.06	0.08	0.14	0.03
Thompson Island Dam	15	3	11	0.05	0.07	0.08	0.01
Schuylerville (Transect)	15	4	11	0.06	0.06	0.07	0.00
Stillwater	15	2	12	0.05	0.09	0.12	0.03
Waterford	15	3	50	0.05	0.10	0.18	0.02
TAL - Arsenic							
Bakers Falls	15	2	24	0.20	0.30	0.40	0.05
Rogers Island	15	1	25	0.21	0.28	0.34	0.03
Thompson Island Dam	15	3	33	0.21	0.32	0.38	0.03
Schuylerville (Transect)	15	4	37	0.20	0.33	0.51	0.04
Stillwater	15	2	24	0.19	0.36	0.53	0.07
Waterford	15	3	56	0.21	0.35	0.77	0.05
TAL - Barium							
Bakers Falls	15	2	100	6.50	8.06	10.70	0.33
Rogers Island	15	1	100	6.60	8.33	11.00	0.34
Thompson Island Dam	15	3	100	6.70	8.60	10.90	0.28
Schuylerville (Transect)	15	4	100	8.00	9.73	14.30	0.37
Stillwater	15	2	100	8.80	10.96	13.80	0.32
Waterford	15	3	100	10.70	13.16	17.00	0.44

**Table 4-4. Baseline water program total TAL metals summary statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Beryllium							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	0	--	--	--	--
Thompson Island Dam	15	3	0	--	--	--	--
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	0	--	--	--	--
Waterford	15	3	0	--	--	--	--
TAL - Cadmium							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	0	--	--	--	--
Thompson Island Dam	15	3	0	--	--	--	--
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	0	--	--	--	--
Waterford	15	3	0	--	--	--	--
TAL - Calcium							
Bakers Falls	15	2	100	5190.00	7305.88	11200.00	486.68
Rogers Island	15	1	100	4860.00	7103.13	11000.00	495.96
Thompson Island Dam	15	3	100	5170.00	7596.67	11500.00	423.44
Schuylerville (Transect)	15	4	100	5410.00	9200.53	20300.00	812.60
Stillwater	15	2	100	7570.00	10281.18	15000.00	459.00
Waterford	15	3	100	8850.00	13991.67	21200.00	881.55
TAL - Chromium							
Bakers Falls	15	2	12	0.34	0.37	0.40	0.03
Rogers Island	15	1	6	0.39	0.39	0.39	--
Thompson Island Dam	15	3	6	0.39	0.39	0.39	--
Schuylerville (Transect)	15	4	5	0.45	0.45	0.45	--
Stillwater	15	2	6	0.44	0.44	0.44	--
Waterford	15	3	6	0.45	0.45	0.45	--

**Table 4-4. Baseline water program total TAL metals summary statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Cobalt							
Bakers Falls	15	2	88	0.03	0.06	0.08	0.00
Rogers Island	15	1	88	0.03	0.08	0.18	0.01
Thompson Island Dam	15	3	83	0.03	0.08	0.16	0.01
Schuylerville (Transect)	15	4	95	0.04	0.09	0.16	0.01
Stillwater	15	2	94	0.04	0.12	0.28	0.02
Waterford	15	3	94	0.03	0.16	0.43	0.03
TAL - Copper							
Bakers Falls	15	2	6	1.10	1.10	1.10	--
Rogers Island	15	1	13	3.90	5.55	7.20	1.65
Thompson Island Dam	15	3	11	1.50	1.80	2.10	0.30
Schuylerville (Transect)	15	4	5	1.20	1.20	1.20	--
Stillwater	15	2	24	1.20	2.73	6.20	1.17
Waterford	15	3	28	1.40	2.00	3.00	0.28
TAL - Iron							
Bakers Falls	15	2	100	102.00	169.00	268.00	9.62
Rogers Island	15	1	100	94.70	198.86	371.00	19.03
Thompson Island Dam	15	3	100	127.00	224.50	429.00	17.36
Schuylerville (Transect)	15	4	100	120.00	251.58	430.00	20.91
Stillwater	15	2	100	142.00	310.00	842.00	42.10
Waterford	15	3	100	46.20	364.84	1050.00	57.02
TAL - Lead							
Bakers Falls	15	2	76	0.06	0.13	0.21	0.01
Rogers Island	15	1	81	0.06	0.15	0.27	0.02
Thompson Island Dam	15	3	83	0.08	0.23	0.42	0.03
Schuylerville (Transect)	15	4	84	0.12	0.31	0.55	0.04
Stillwater	15	2	82	0.10	0.38	0.88	0.07
Waterford	15	3	83	0.13	0.47	1.20	0.08

**Table 4-4. Baseline water program total TAL metals summary statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Magnesium							
Bakers Falls	15	2	100	730.00	1065.00	1530.00	53.67
Rogers Island	15	1	100	774.00	1058.25	1540.00	50.88
Thompson Island Dam	15	3	100	969.00	1209.61	1600.00	42.88
Schuylerville (Transect)	15	4	100	1070.00	1697.37	3270.00	125.98
Stillwater	15	2	100	1640.00	2056.47	2740.00	68.24
Waterford	15	3	100	1980.00	2754.44	3890.00	133.19
TAL - Manganese							
Bakers Falls	15	2	100	20.90	34.14	69.90	3.98
Rogers Island	15	1	100	20.10	33.93	76.40	3.88
Thompson Island Dam	15	3	100	22.00	32.95	51.40	2.61
Schuylerville (Transect)	15	4	100	20.80	32.87	48.00	1.66
Stillwater	15	2	100	20.00	34.95	54.00	2.40
Waterford	15	3	100	13.60	37.59	68.50	3.26
TAL - Mercury							
Bakers Falls	15	2	6	0.09	0.09	0.09	--
Rogers Island	15	1	6	0.08	0.08	0.08	--
Thompson Island Dam	15	3	6	0.06	0.06	0.06	--
Schuylerville (Transect)	15	4	5	0.05	0.05	0.05	--
Stillwater	15	2	0	--	--	--	--
Waterford	15	3	17	0.05	0.06	0.06	0.00
TAL - Nickel							
Bakers Falls	15	2	88	0.14	0.24	0.32	0.01
Rogers Island	15	1	88	0.14	0.27	0.38	0.02
Thompson Island Dam	15	3	89	0.18	0.31	0.40	0.02
Schuylerville (Transect)	15	4	89	0.11	0.30	0.42	0.02
Stillwater	15	2	94	0.17	0.34	0.58	0.03
Waterford	15	3	94	0.27	0.46	0.86	0.04

**Table 4-4. Baseline water program total TAL metals summary statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Potassium							
Bakers Falls	15	2	94	298.00	467.44	739.00	36.69
Rogers Island	15	1	94	298.00	451.73	703.00	34.91
Thompson Island Dam	15	3	94	325.00	486.35	711.00	30.00
Schuylerville (Transect)	15	4	95	344.00	503.17	713.00	27.88
Stillwater	15	2	88	398.00	584.27	764.00	31.40
Waterford	15	3	94	527.00	713.47	964.00	34.14
TAL - Selenium							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	6	0.41	0.41	0.41	--
Thompson Island Dam	15	3	6	0.63	0.63	0.63	--
Schuylerville (Transect)	15	4	11	0.25	0.39	0.53	0.14
Stillwater	15	2	12	0.33	0.41	0.49	0.08
Waterford	15	3	6	0.65	0.65	0.65	--
TAL - Silver							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	0	--	--	--	--
Thompson Island Dam	15	3	0	--	--	--	--
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	0	--	--	--	--
Waterford	15	3	0	--	--	--	--
TAL - Sodium							
Bakers Falls	15	2	100	3420.00	5617.65	8940.00	422.32
Rogers Island	15	1	100	3360.00	5371.25	8840.00	423.96
Thompson Island Dam	15	3	100	3500.00	5775.56	8980.00	388.92
Schuylerville (Transect)	15	4	100	3500.00	5679.47	10200.00	436.93
Stillwater	15	2	100	4750.00	6482.35	9940.00	344.47
Waterford	15	3	100	5270.00	8396.11	12300.00	541.72

**Table 4-4. Baseline water program total TAL metals summary statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Thallium							
Bakers Falls	15	2	12	0.07	0.11	0.16	0.05
Rogers Island	15	1	19	0.06	0.14	0.19	0.04
Thompson Island Dam	15	3	17	0.08	0.13	0.21	0.04
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	6	0.08	0.08	0.08	--
Waterford	15	3	56	0.06	0.09	0.21	0.01
TAL - Vanadium							
Bakers Falls	15	2	18	0.50	0.81	1.10	0.17
Rogers Island	15	1	13	1.30	1.40	1.50	0.10
Thompson Island Dam	15	3	17	0.62	1.04	1.30	0.21
Schuylerville (Transect)	15	4	16	0.60	1.03	1.50	0.26
Stillwater	15	2	12	1.10	10.40	19.70	9.30
Waterford	15	3	11	0.31	0.91	1.50	0.60
TAL - Zinc							
Bakers Falls	15	2	18	2.40	4.50	5.80	1.06
Rogers Island	15	1	19	2.20	3.60	6.20	1.30
Thompson Island Dam	15	3	17	3.60	5.53	6.80	0.98
Schuylerville (Transect)	15	4	16	2.40	5.27	6.80	1.43
Stillwater	15	2	6	1.70	1.70	1.70	--
Waterford	15	3	17	3.10	15.97	29.60	7.66

*Note:*

*Statistics based on detectable concentrations only.*

**Table 4-5. Baseline Water Program Dissolved TAL Metals Summary Statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Aluminum (DISS)							
Bakers Falls	15	2	82	17.2	40.63	82.50	4.84
Rogers Island	15	1	81	10.4	47.51	178.00	11.66
Thompson Island Dam	15	3	72	12.7	38.44	84.50	5.48
Schuylerville (Transect)	15	4	79	11.3	39.46	82.90	5.69
Stillwater	15	2	82	9.3	34.74	63.30	4.61
Waterford	15	3	78	9.4	35.12	68.80	4.64
TAL - Antimony (DISS)							
Bakers Falls	15	2	24	0.053	0.07	0.08	0.01
Rogers Island	15	1	31	0.05	0.09	0.17	0.02
Thompson Island Dam	15	3	39	0.036	0.08	0.18	0.02
Schuylerville (Transect)	15	4	32	0.068	0.10	0.23	0.03
Stillwater	15	2	35	0.058	0.13	0.35	0.05
Waterford	15	3	39	0.069	0.13	0.22	0.02
TAL - Arsenic (DISS)							
Bakers Falls	15	2	35	0.21	0.31	0.48	0.04
Rogers Island	15	1	31	0.23	0.31	0.47	0.04
Thompson Island Dam	15	3	33	0.19	0.24	0.34	0.03
Schuylerville (Transect)	15	4	26	0.19	0.29	0.45	0.04
Stillwater	15	2	29	0.18	0.32	0.52	0.07
Waterford	15	3	39	0.2	0.33	0.51	0.05
TAL - Barium (DISS)							
Bakers Falls	15	2	100	6.3	7.75	10.20	0.35
Rogers Island	15	1	100	6	7.70	10.40	0.36
Thompson Island Dam	15	3	100	5.9	7.88	10.60	0.33
Schuylerville (Transect)	15	4	100	6.7	8.81	12.50	0.38
Stillwater	15	2	100	8.4	9.50	12.20	0.29
Waterford	15	3	100	8.6	11.74	15.50	0.49

**Table 4-5. Baseline Water Program Dissolved TAL Metals Summary Statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Beryllium (DISS)							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	0	--	--	--	--
Thompson Island Dam	15	3	0	--	--	--	--
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	0	--	--	--	--
Waterford	15	3	0	--	--	--	--
TAL - Cadmium (DISS)							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	6	0.13	0.13	0.13	--
Thompson Island Dam	15	3	0	--	--	--	--
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	0	--	--	--	--
Waterford	15	3	0	--	--	--	--
TAL - Calcium (DISS)							
Bakers Falls	15	2	100	5060	7370.00	11400.00	510.53
Rogers Island	15	1	100	4960	7066.88	10800.00	487.15
Thompson Island Dam	15	3	100	5090	7520.56	10900.00	417.55
Schuylerville (Transect)	15	4	100	5440	8756.32	12800.00	528.82
Stillwater	15	2	100	7780	10087.06	14900.00	442.48
Waterford	15	3	100	8800	14541.11	22200.00	987.63
TAL - Chromium (DISS)							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	0	--	--	--	--
Thompson Island Dam	15	3	0	--	--	--	--
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	0	--	--	--	--
Waterford	15	3	0	--	--	--	--

**Table 4-5. Baseline Water Program Dissolved TAL Metals Summary Statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Cobalt (DISS)							
Bakers Falls	15	2	47	0.045	0.92	1.90	0.29
Rogers Island	15	1	38	0.044	0.30	0.80	0.15
Thompson Island Dam	15	3	44	0.049	0.88	1.90	0.26
Schuylerville (Transect)	15	4	53	0.032	0.62	1.50	0.19
Stillwater	15	2	53	0.037	0.78	1.80	0.23
Waterford	15	3	50	0.038	0.97	2.30	0.26
TAL - Copper (DISS)							
Bakers Falls	15	2	6	5.7	5.70	5.70	--
Rogers Island	15	1	6	1.8	1.80	1.80	--
Thompson Island Dam	15	3	11	2.1	5.25	8.40	3.15
Schuylerville (Transect)	15	4	5	1.3	1.30	1.30	--
Stillwater	15	2	6	22.3	22.30	22.30	--
Waterford	15	3	11	1.7	3.75	5.80	2.05
TAL - Iron (DISS)							
Bakers Falls	15	2	94	38.9	81.80	177.00	8.45
Rogers Island	15	1	94	34.4	87.87	265.00	14.25
Thompson Island Dam	15	3	94	45.6	82.24	114.00	5.82
Schuylerville (Transect)	15	4	95	30.4	82.04	124.00	7.04
Stillwater	15	2	88	39.3	82.19	124.00	7.62
Waterford	15	3	94	29.1	88.30	159.00	9.39
TAL - Lead (DISS)							
Bakers Falls	15	2	41	0.025	0.06	0.10	0.01
Rogers Island	15	1	50	0.026	0.12	0.44	0.06
Thompson Island Dam	15	3	56	0.031	0.06	0.12	0.01
Schuylerville (Transect)	15	4	53	0.039	0.09	0.36	0.03
Stillwater	15	2	53	0.023	0.08	0.24	0.02
Waterford	15	3	50	0.043	0.10	0.15	0.01

**Table 4-5. Baseline Water Program Dissolved TAL Metals Summary Statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Magnesium (DISS)							
Bakers Falls	15	2	100	726	1077.82	1540.00	56.31
Rogers Island	15	1	100	752	1047.88	1540.00	50.16
Thompson Island Dam	15	3	100	930	1194.89	1560.00	45.39
Schuylerville (Transect)	15	4	100	1060	1640.53	2490.00	93.43
Stillwater	15	2	100	1650	2020.59	2670.00	69.31
Waterford	15	3	100	1910	2774.44	3960.00	146.16
TAL - Manganese (DISS)							
Bakers Falls	15	2	100	3.9	19.92	50.30	3.29
Rogers Island	15	1	100	5	16.01	39.60	2.49
Thompson Island Dam	15	3	100	7.4	19.96	42.40	2.68
Schuylerville (Transect)	15	4	100	9.3	18.49	30.20	1.62
Stillwater	15	2	100	9.6	18.11	30.90	1.28
Waterford	15	3	100	5.5	15.51	32.10	1.47
TAL - Mercury (DISS)							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	0	--	--	--	--
Thompson Island Dam	15	3	6	0.055	0.06	0.06	--
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	12	0.049	0.06	0.07	0.01
Waterford	15	3	0	--	--	--	--
TAL - Nickel (DISS)							
Bakers Falls	15	2	47	0.19	0.41	0.62	0.05
Rogers Island	15	1	56	0.2	0.33	0.49	0.03
Thompson Island Dam	15	3	61	0.18	0.39	0.52	0.04
Schuylerville (Transect)	15	4	53	0.21	1.28	9.30	0.89
Stillwater	15	2	59	0.21	0.47	1.00	0.07
Waterford	15	3	50	0.33	0.47	0.67	0.04

**Table 4-5. Baseline Water Program Dissolved TAL Metals Summary Statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Potassium (DISS)							
Bakers Falls	15	2	88	308	480.00	752.00	38.55
Rogers Island	15	1	94	308	444.13	687.00	34.14
Thompson Island Dam	15	3	89	322	482.75	682.00	31.26
Schuylerville (Transect)	15	4	84	349	507.81	727.00	27.43
Stillwater	15	2	88	408	560.93	737.00	29.23
Waterford	15	3	94	481	707.12	965.00	37.70
TAL - Selenium (DISS)							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	6	0.28	0.28	0.28	--
Thompson Island Dam	15	3	0	--	--	--	--
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	0	--	--	--	--
Waterford	15	3	0	--	--	--	--
TAL - Silver (DISS)							
Bakers Falls	15	2	0	--	--	--	--
Rogers Island	15	1	0	--	--	--	--
Thompson Island Dam	15	3	0	--	--	--	--
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	0	--	--	--	--
Waterford	15	3	0	--	--	--	--
TAL - Sodium (DISS)							
Bakers Falls	15	2	100	3500	5675.29	8950.00	435.06
Rogers Island	15	1	100	3350	5316.88	8620.00	414.86
Thompson Island Dam	15	3	100	3370	5755.00	8620.00	392.01
Schuylerville (Transect)	15	4	100	3390	5557.89	8870.00	382.76
Stillwater	15	2	100	5060	6362.94	9660.00	327.46
Waterford	15	3	100	5420	8605.56	12800.00	571.77

**Table 4-5. Baseline Water Program Dissolved TAL Metals Summary Statistics.**

Location	Sample Counts		Frequency Detected (%)	TAL Metals (µg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
TAL - Thallium (DISS)							
Bakers Falls	15	2	6	0.14	0.14	0.14	--
Rogers Island	15	1	0	--	--	--	--
Thompson Island Dam	15	3	11	0.12	0.18	0.23	0.06
Schuylerville (Transect)	15	4	0	--	--	--	--
Stillwater	15	2	12	0.074	0.14	0.20	0.06
Waterford	15	3	28	0.058	0.09	0.14	0.02
TAL - Vanadium (DISS)							
Bakers Falls	15	2	12	0.63	0.82	1.00	0.19
Rogers Island	15	1	13	0.94	0.94	0.94	0.00
Thompson Island Dam	15	3	6	0.51	0.51	0.51	--
Schuylerville (Transect)	15	4	11	0.24	0.82	1.40	0.58
Stillwater	15	2	12	0.32	0.81	1.30	0.49
Waterford	15	3	6	1	1.00	1.00	--
TAL - Zinc (DISS)							
Bakers Falls	15	2	6	3.4	3.40	3.40	--
Rogers Island	15	1	19	2.1	9.33	23.50	7.08
Thompson Island Dam	15	3	11	4.5	14.45	24.40	9.95
Schuylerville (Transect)	15	4	11	2.3	7.45	12.60	5.15
Stillwater	15	2	12	2	13.55	25.10	11.55
Waterford	15	3	11	3.2	10.20	17.20	7.00

*Notes:*

*Statistics based on detectable concentrations only.*

**Table 4-6. Baseline water quality parameter summary statistics.**

Location	Specific Conductance			Temperature			Turbidity			pH			Dissolved Oxygen		
	Min	Avg	Max	Min	Avg	Max	Min	Avg	Max	Min	Avg	Max	Min	Avg	Max
Bakers Falls	0.047	0.078	0.13	0.3	10.4	26.0	0.0	1.4	11.6	6.52	7.42	8.27	4.34	12.14	19.81
Rogers Island	0.051	0.081	0.13	0.3	12.3	25.1	0.0	1.4	5.3	6.35	7.38	8.69	5.69	11.26	23.80
Thompson Island (PRW2)	0.067	0.068	0.07	11.9	12.0	12.1	2.4	3.1	3.6	6.88	7.01	7.10	NC	NC	NC
Thompson Island Dam	0.053	0.088	0.148	5.4	15.7	25.3	0.0	6.5	935.3	6.75	7.42	8.31	0.57	10.66	38.80
Schuylerville (Transect)	0.055	0.099	0.219	0.2	11.9	25.5	0.0	8.4	875.7	6.04	7.48	8.51	2.57	11.88	59.17
Schuylerville (Center)	0.072	0.076	0.092	12.3	12.4	12.4	3.6	4.7	7.2	6.86	7.00	7.13	NC	NC	NC
Stillwater	0.084	0.121	0.211	5.5	17.7	26.5	0.0	6.1	67.0	6.41	7.52	7.97	3.51	9.92	15.60
Waterford	0.087	0.144	0.293	0.3	11.9	26.4	0.1	35.6	925.2	6.13	7.66	9.09	2.24	11.30	24.35
Mohawk River at Cohoes	0.121	0.279	0.376	3.1	14.1	24.0	4.3	63.4	862.0	7.35	7.92	8.34	2.30	10.79	14.84
LHR Albany	0.172	0.216	0.269	8.2	17.6	24.2	2.8	15.8	29.5	7.56	7.97	8.30	8.15	10.66	12.39
LHR Poughkeepsie	0.178	0.220	0.267	9.1	19.6	26.6	10.9	15.7	19.3	7.43	7.84	8.22	7.17	8.75	10.59

Notes:

NC - Not collected.

**Table 5-1. Aroclor PCB summary statistics for black bass.**

Species	Pool	Station Number	Count	Average mg/kg	Minimum mg/kg	Maximum mg/kg	2 SE mg/kg
Largemouth bass	Feeder Dam	1	14	0.05	ND	0.13	0.02
	Thompson Island Pool	2	1	1.73	1.73	1.73	-
	Thompson Island Pool	3	1	4.25	4.25	4.25	-
	Thompson Island Pool	4	1	2.54	2.54	2.54	-
	Thompson Island Pool	5	10	2.44	0.13	7.08	1.53
	Northumberland/Fort Miller	3	1	3.17	3.17	3.17	-
	Northumberland/Fort Miller	5	10	2.24	0.28	6.46	1.26
	Stillwater	1	1	7.29	7.29	7.29	-
	Stillwater	3	10	2.07	0.13	6.38	1.37
	Albany/Troy	1	1	0.09	0.09	0.09	-
Smallmouth bass	Feeder Dam	1	6	0.03	ND	0.06	0.01
	Thompson Island Pool	1	5	1.38	0.93	1.78	0.38
	Thompson Island Pool	2	4	1.77	0.38	5.04	2.22
	Thompson Island Pool	3	4	4.09	2.97	6.95	1.91
	Thompson Island Pool	4	4	1.44	0.19	2.86	1.33
	Northumberland/Fort Miller	1	5	1.60	1.10	2.17	0.39
	Northumberland/Fort Miller	2	5	1.69	0.49	3.07	1.07
	Northumberland/Fort Miller	3	4	2.43	0.80	3.73	1.21
	Stillwater	1	4	4.10	3.01	5.46	1.23
	Stillwater	2	5	1.90	0.63	3.55	0.96
	Stillwater	4	5	0.95	0.32	1.61	0.44
	Stillwater	5	5	0.95	0.53	2.41	0.73
	Albany/Troy	1	20	1.83	0.08	6.31	0.68

Notes:

Prep: fillet

Non-detect values set to half method detection limit to calculate average and 2 SE.

ND = Non Detect

**Table 5-2. Congener-specific PCB summary statistics for black bass.**

Species	Pool	Station Number	Count	Average mg/kg	Minimum mg/kg	Maximum mg/kg	2 SE mg/kg
Largemouth bass	Feeder Dam	1	2	0.02	0.02	0.02	0.01
	Thompson Island Pool	4	1	1.74	1.74	1.74	-
	Northumberland/Fort Miller	5	1	4.08	4.08	4.08	-
	Stillwater	3	1	0.09	0.09	0.09	-
	Albany/Troy	1	1	0.14	0.14	0.14	-
Smallmouth bass	Thompson Island Pool	1	1	0.62	0.62	0.62	-
	Northumberland/Fort Miller	1	1	1.02	1.02	1.02	-
	Northumberland/Fort Miller	2	1	2.14	2.14	2.14	-
	Stillwater	1	1	3.33	3.33	3.33	-
	Stillwater	5	1	0.53	0.53	0.53	-
	Albany/Troy	1	2	1.27	0.84	1.71	0.87

Notes:

Prep = fillet

**Table 5-3. Aroclor PCB summary statistics for ictalurids.**

Species	Pool	Station Number	Count	Average mg/kg	Minimum mg/kg	Maximum mg/kg	2 SE mg/kg
Brown bullhead	Feeder Dam	1	17	0.14	ND	0.67	0.09
	Thompson Island Pool	1	3	1.95	0.51	3.06	1.51
	Thompson Island Pool	2	5	2.80	1.17	3.96	1.11
	Thompson Island Pool	3	5	6.59	2.45	16.97	5.69
	Thompson Island Pool	4	5	1.29	0.48	3.34	1.04
	Thompson Island Pool	5	10	5.24	0.52	10.13	2.13
	Northumberland/Fort Miller	1	5	5.05	3.42	6.44	1.06
	Northumberland/Fort Miller	2	1	3.76	3.76	3.76	-
	Northumberland/Fort Miller	3	5	4.43	2.41	7.08	1.92
	Northumberland/Fort Miller	5	10	3.58	0.77	7.03	1.15
	Stillwater	1	4	2.65	1.77	3.83	0.96
	Stillwater	2	4	1.72	1.33	2.26	0.39
	Stillwater	3	10	3.50	0.23	7.44	1.61
	Stillwater	4	5	4.42	3.30	6.16	1.08
	Stillwater	5	5	7.08	3.30	16.10	4.69
Channel catfish	Albany/Troy	1	3	5.21	4.14	5.90	1.08
White catfish	Albany/Troy	1	17	2.94	1.15	5.35	0.48
Yellow bullhead	Feeder Dam	1	3	0.02	ND	0.04	0.02
	Thompson Island Pool	1	2	6.10	5.59	6.61	1.03
	Northumberland/Fort Miller	2	1	3.50	3.50	3.50	-
	Stillwater	1	1	3.03	3.03	3.03	-
	Stillwater	2	1	0.43	0.43	0.43	-

Notes:

Prep: fillet

Non-detect values set to half method detection limit to calculate average and 2 SE.

ND = Non Detect

**Table 5-4. Congener-specific PCB summary statistics for ictalurids.**

Species	Pool	Station Number	Count	Average mg/kg	Minimum mg/kg	Maximum mg/kg	2 SE mg/kg
Brown bullhead	Feeder Dam	1	3	0.06	0.02	0.08	0.04
	Thompson Island Pool	2	1	2.50	2.50	2.50	-
	Thompson Island Pool	3	1	12.70	12.70	12.70	-
	Northumberland/Fort Miller	2	1	3.14	3.14	3.14	-
	Northumberland/Fort Miller	5	1	1.05	1.05	1.05	-
	Stillwater	2	1	2.26	2.26	2.26	-
	Stillwater	3	1	2.40	2.40	2.40	-
Channel catfish	Albany/Troy	1	1	4.31	4.31	4.31	-
White catfish	Albany/Troy	1	1	1.97	1.97	1.97	-
Yellow bullhead	Thompson Island Pool	1	1	3.60	3.60	3.60	-
	Stillwater	2	1	0.32	0.32	0.32	-

Notes:

Prep = fillet

**Table 5-5. Aroclor PCB summary statistics for perch.**

Species	Pool	Station Number	Count	Average mg/kg	Minimum mg/kg	Maximum mg/kg	2 SE mg/kg
White perch	Albany/Troy	1	20	0.71	0.37	1.45	0.12
Yellow perch	Feeder Dam	1	20	0.02	ND	0.10	0.01
	Thompson Island Pool	1	5	1.38	0.72	2.40	0.62
	Thompson Island Pool	2	5	0.86	0.54	1.41	0.33
	Thompson Island Pool	3	5	4.64	2.75	9.23	2.36
	Thompson Island Pool	4	5	0.47	0.41	0.61	0.07
	Thompson Island Pool	5	10	1.16	0.52	2.16	0.35
	Northumberland/Fort Miller	1	5	1.10	0.23	2.57	0.82
	Northumberland/Fort Miller	2	6	1.84	0.81	4.21	1.03
	Northumberland/Fort Miller	3	5	1.23	0.47	2.27	0.59
	Northumberland/Fort Miller	5	10	1.09	0.47	1.43	0.20
	Stillwater	1	5	0.45	0.08	0.91	0.32
	Stillwater	2	5	0.67	0.19	1.26	0.37
	Stillwater	3	10	1.12	0.39	1.88	0.27
	Stillwater	4	5	0.37	0.16	0.60	0.17
	Stillwater	5	5	0.24	0.17	0.28	0.04

Notes:

Prep: fillet

Non-detect values set to half method detection limit to calculate average and 2 SE.

ND = Non Detect

**Table 5-6. Congener-specific PCB summary statistics for perch.**

Species	Pool	Station Number	Count	Average mg/kg	Minimum mg/kg	Maximum mg/kg	2 SE mg/kg
White perch	Albany/Troy	1	2	0.54	0.50	0.58	0.08
Yellow perch	Feeder Dam	1	1	0.02	0.02	0.02	-
	Thompson Island	1	1	1.50	1.50	1.50	-
	Thompson Island	3	1	2.76	2.76	2.76	-
	Thompson Island	5	1	0.64	0.64	0.64	-
	Northumberland/Fort Miller	2	1	1.44	1.44	1.44	-
	Northumberland/Fort Miller	3	1	1.19	1.19	1.19	-
	Northumberland/Fort Miller	5	1	1.27	1.27	1.27	-
	Stillwater	2	1	0.27	0.27	0.27	-
	Stillwater	5	1	0.26	0.26	0.26	-

Notes:

Prep = fillet

**Table 5-7. Aroclor PCB summary statistics for pumpkinseed.**

Pool	Station Number	Count	Average mg/kg	Minimum mg/kg	Maximum mg/kg	2 SE mg/kg
Feeder Dam	1	20	0.08	0.01	0.44	0.04
Thompson Island Pool	1	5	4.77	2.17	9.51	2.64
Thompson Island Pool	2	5	4.72	3.57	7.00	1.40
Thompson Island Pool	3	5	22.04	9.29	44.00	11.64
Thompson Island Pool	4	5	5.64	2.85	10.49	2.56
Thompson Island Pool	5	10	7.25	4.14	14.70	2.03
Northumberland/Fort Miller	3	10	3.29	1.17	11.63	1.99
Northumberland/Fort Miller	5	15	9.18	4.16	18.15	2.52
Stillwater	1	5	4.52	2.98	7.33	1.48
Stillwater	2	5	2.74	1.07	4.35	1.07
Stillwater	3	5	1.95	1.23	3.30	0.76
Stillwater	4	5	2.10	1.68	2.64	0.34
Stillwater	5	10	2.46	1.37	3.11	0.361
Albany/Troy	1	20	0.79	0.44	1.20	0.09

*Notes:*

*Prep: whole body*

*Non-detect values set to half method detection limit to calculate average and 2 SE.*

*ND = Non Detect*

**Table 5-8. Congener-specific PCB summary statistics for pumpkinseed.**

<b>Pool</b>	<b>Station Number</b>	<b>Count</b>	<b>Average mg/kg</b>	<b>Minimum mg/kg</b>	<b>Maximum mg/kg</b>	<b>2 SE mg/kg</b>
Thompson Island Pool	1	1	1.69	1.69	1.69	-
Thompson Island Pool	2	1	4.22	4.22	4.22	-
Thompson Island Pool	4	1	3.65	3.65	3.65	-
Thompson Island Pool	5	1	10.80	10.80	10.80	-
Northumberland/Fort Miller	3	1	1.33	1.33	1.33	-
Stillwater	2	1	2.49	2.49	2.49	-
Stillwater	5	1	2.01	2.01	2.01	-

*Notes:*

*Prep = whole body*

**Table 5-9. Aroclor PCB summary statistics for forage fish.**

Pool	Station Number	Count	Average mg/kg	Minimum mg/kg	Maximum mg/kg	2 SE mg/kg
Feeder Dam	1	10	0.09	0.03	0.17	0.03
Thompson Island Pool	1	2	5.09	3.13	7.06	3.93
Thompson Island Pool	2	2	5.70	4.08	7.33	3.25
Thompson Island Pool	3	2	10.60	8.21	12.99	4.78
Thompson Island Pool	4	2	4.00	3.55	4.44	0.89
Thompson Island Pool	5	2	9.93	8.87	10.99	2.12
Northumberland/Fort Miller	3	5	8.36	5.40	16.16	4.01
Northumberland/Fort Miller	5	5	4.04	2.62	6.47	1.41
Stillwater	1	2	4.48	4.09	4.87	0.78
Stillwater	2	2	2.50	2.17	2.84	0.67
Stillwater	3	2	2.53	2.49	2.56	0.07
Stillwater	4	2	1.23	0.85	1.62	0.76
Stillwater	5	2	4.50	3.55	5.45	1.90
Albany/Troy	1	3	1.21	0.91	1.52	0.35

Notes:

Prep: whole body (composite)

Non-detect values set to half method detection limit to calculate average and 2 SE.

ND = Non Detect

**Table 5-10. Congener-specific PCB summary statistics for forage fish.**

Pool	Station Number	Count	Average mg/kg	Minimum mg/kg	Maximum mg/kg	2 SE mg/kg
Feeder Dam	1	1	0.05	0.05	0.05	-
Thompson Island Pool	2	1	2.85	2.85	2.85	-
Thompson Island Pool	3	1	8.76	8.76	8.76	-
Thompson Island Pool	4	1	2.94	2.94	2.94	-
Thompson Island Pool	5	1	6.49	6.49	6.49	-
Northumberland/Fort Miller	5	1	2.74	2.74	2.74	-
Stillwater	1	1	3.47	3.47	3.47	-
Stillwater	2	1	2.28	2.28	2.28	-
Stillwater	3	1	2.11	2.11	2.11	-
Stillwater	4	1	0.66	0.66	0.66	-

Notes:

*Prep=whole body (composite)*

**Table 5-11. Percent lipid summary statistics for black bass.**

Species	Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
Largemouth bass	Feeder Dam	1	14	0.35	0.11	1.05	0.14
	Thompson Island Dam	2	1	0.34	0.34	0.34	-
	Thompson Island Dam	3	1	0.42	0.42	0.42	-
	Thompson Island Dam	4	1	0.05	0.05	0.05	-
	Thompson Island Dam	5	10	0.42	0.05	0.79	0.15
	Northumberland/Fort Miller	3	1	0.28	0.28	0.28	-
	Northumberland/Fort Miller	5	10	0.47	0.08	1.07	0.21
	Stillwater	1	1	1.03	1.03	1.03	-
	Stillwater	3	10	0.47	0.10	1.20	0.22
	Albany/Troy	1	1	1.00	1.00	1.00	-
Smallmouth bass	Feeder Dam	1	6	0.25	0.13	0.38	0.07
	Thompson Island Dam	1	5	0.44	0.21	0.76	0.18
	Thompson Island Dam	2	4	0.70	0.41	0.92	0.23
	Thompson Island Dam	3	4	0.52	0.42	0.62	0.08
	Thompson Island Dam	4	4	0.39	0.08	0.55	0.22
	Northumberland/Fort Miller	1	5	0.51	0.39	0.61	0.07
	Northumberland/Fort Miller	2	5	0.63	0.46	0.70	0.09
	Northumberland/Fort Miller	3	4	0.45	0.28	0.66	0.17
	Stillwater	1	4	0.87	0.42	1.73	0.59
	Stillwater	2	5	0.75	0.47	1.29	0.30
	Stillwater	4	5	0.55	0.25	1.09	0.29
	Stillwater	5	5	0.31	0.13	0.61	0.16
	Albany/Troy	1	20	0.71	0.25	1.86	0.22

Notes:

Prep=fillet

**Table 5-12. Percent lipid summary statistics for ictalurids.**

Species	Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
Brown bullhead	Feeder Dam	1	17	0.95	0.29	2.81	0.29
	Thompson Island Pool	1	3	1.33	1.05	1.77	0.45
	Thompson Island Pool	2	5	2.00	1.36	2.89	0.60
	Thompson Island Pool	3	5	2.14	0.97	4.97	1.47
	Thompson Island Pool	4	5	1.06	0.43	1.90	0.58
	Thompson Island Pool	5	10	1.74	0.57	3.70	0.68
	Northumberland/Fort Miller	1	5	1.91	1.19	2.62	0.56
	Northumberland/Fort Miller	2	1	2.83	2.83	2.83	-
	Northumberland/Fort Miller	3	5	1.85	1.16	3.53	0.88
	Northumberland/Fort Miller	5	10	1.76	0.90	3.17	0.48
	Stillwater	1	4	1.83	1.09	3.02	0.88
	Stillwater	2	4	1.58	0.66	2.24	0.71
	Stillwater	3	10	2.44	0.31	6.69	1.13
	Stillwater	4	5	3.29	1.80	4.25	0.81
	Stillwater	5	5	4.49	1.45	9.83	2.90
Channel catfish	Albany/Troy	1	3	9.08	7.58	11.80	2.72
White catfish	Albany/Troy	1	17	4.01	1.61	7.54	0.78
Yellow bullhead	Feeder Dam	1	3	1.03	0.27	2.42	1.39
	Thompson Island Pool	1	2	1.02	0.89	1.14	0.25
	Northumberland/Fort Miller	2	1	0.76	0.76	0.76	-
	Stillwater	1	1	1.63	1.63	1.63	-
	Stillwater	2	1	0.63	0.63	0.63	-

Notes:

Prep=fillet

**Table 5-13. Percent lipid summary statistics for perch.**

Species	Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
White perch	Albany/Troy	1	20	0.80	0.33	1.99	0.21
Yellow perch	Feeder Dam	1	20	0.67	0.21	1.04	0.10
	Thompson Island Pool	1	5	0.60	0.26	1.04	0.26
	Thompson Island Pool	2	5	0.82	0.29	1.63	0.49
	Thompson Island Pool	3	5	0.93	0.60	1.16	0.26
	Thompson Island Pool	4	5	0.33	0.10	0.81	0.25
	Thompson Island Pool	5	10	0.97	0.43	1.72	0.22
	Northumberland/Fort Miller	1	5	0.68	0.31	1.65	0.49
	Northumberland/Fort Miller	2	6	1.04	0.63	1.67	0.32
	Northumberland/Fort Miller	3	5	0.66	0.28	1.29	0.34
	Northumberland/Fort Miller	5	10	0.89	0.54	1.23	0.14
	Stillwater	1	5	0.56	0.37	0.97	0.22
	Stillwater	2	5	1.10	0.51	1.70	0.43
	Stillwater	3	10	1.00	0.34	2.21	0.31
	Stillwater	4	5	1.05	0.66	1.62	0.35
	Stillwater	5	5	0.48	0.32	0.57	0.09

Notes:

Prep=fillet

**Table 5-14. Percent lipid summary statistics for pumpkinseed.**

Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
Feeder Dam	1	20	2.58	1.67	3.88	0.26
Thompson Island Pool	1	5	2.11	1.71	2.52	0.26
Thompson Island Pool	2	5	2.70	2.14	3.26	0.38
Thompson Island Pool	3	5	3.21	2.80	3.97	0.40
Thompson Island Pool	4	5	2.46	2.01	3.24	0.44
Thompson Island Pool	5	10	2.81	2.40	3.56	0.26
Northumberland/Fort Miller	3	10	3.14	1.79	4.13	0.48
Northumberland/Fort Miller	5	15	3.12	2.14	5.03	0.36
Stillwater	1	5	3.65	3.31	3.92	0.20
Stillwater	2	5	2.32	1.93	2.57	0.22
Stillwater	3	5	2.87	2.14	3.85	0.79
Stillwater	4	5	2.65	2.20	3.10	0.36
Stillwater	5	10	2.92	2.20	3.94	0.30
Albany/Troy	1	20	2.23	1.28	3.04	0.22

*Notes:*

*Prep: whole body*

**Table 5-15. Percent lipid summary statistics for forage fish.**

Pool	Station Number	Count	Average %	Minimum %	Maximum %	2 SE %
Feeder Dam	1	10	2.45	1.86	3.01	0.25
Thompson Island Pool	1	2	3.18	1.86	4.49	2.63
Thompson Island Pool	2	2	4.46	4.26	4.66	0.40
Thompson Island Pool	3	2	4.27	4.19	4.35	0.16
Thompson Island Pool	4	2	2.59	1.62	3.55	1.93
Thompson Island Pool	5	2	5.09	5.02	5.15	0.13
Northumberland/Fort Miller	3	5	4.92	3.48	6.05	0.94
Northumberland/Fort Miller	5	5	3.40	2.47	4.53	0.71
Stillwater	1	2	5.24	5.11	5.36	0.25
Stillwater	2	2	3.59	3.49	3.68	0.19
Stillwater	3	2	3.24	3.01	3.46	0.45
Stillwater	4	2	2.17	1.61	2.72	1.11
Stillwater	5	2	6.17	4.86	7.48	2.62
Albany/Troy	1	3	4.04	2.16	4.99	1.88

*Notes:*

*Prep: whole body (composite)*

**Table 5-16. Gender summary for black bass.**

Species	Pool	Total Count	Count of Males	Count of Females	Count of Unknowns
Largemouth bass	Feeder Dam	14	6	5	3
	Thompson Island Pool	13	5	7	1
	Northumberland/Fort Miller	11	5	6	0
	Stillwater	11	6	4	1
	Albany/Troy	1	0	1	0
Smallmouth bass	Feeder Dam	6	1	2	3
	Thompson Island Pool	17	5	12	0
	Northumberland/Fort Miller	14	8	6	0
	Stillwater	19	7	12	0
	Albany/Troy	20	9	11	0

Notes:

Prep: fillet

**Table 5-17. Gender summary for ictalurids.**

Species	Pool	Total Count	Count of Males	Count of Females	Count of Unknowns
Brown bullhead	Feeder Dam	17	10	7	0
	Thompson Island Pool	28	7	21	0
	Northumberland/Fort Miller	21	13	8	0
	Stillwater	28	15	13	0
Channel catfish	Albany/Troy	3	3	0	0
White catfish	Albany/Troy	17	10	7	0
Yellow bullhead	Feeder Dam	3	2	1	0
	Thompson Island Pool	2	0	2	0
	Northumberland/Fort Miller	1	1	0	0
	Stillwater	2	1	1	0

Notes:

Prep: fillet

**Table 5-18. Gender summary for perch.**

Species	Pool	Total Count	Count of Males	Count of Females	Count of Unknowns
White perch	Albany/Troy	20	12	5	3
Yellow perch	Feeder Dam	20	11	7	2
	Thompson Island Pool	30	21	5	4
	Northumberland/Fort Miller	26	16	9	1
	Stillwater	30	19	4	7

*Notes:*

*Prep: fillet*

**Table 6-1. Special study summary PCB statistics.**

Location	Sample Counts		Frequency Detected (%)	PCBs (ng/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
Thompson Island (PRW2)	8	0	88	17.82	25.12	31.70	2.22
Schuylerville (Center)	3	0	100	10.70	27.66	43.77	9.55
Waterford High Flow	29	0	100	9.67	51.20	265.00	10.49

*Notes:*

*Statistics based on detectable concentrations only.*

**Table 6-2. Special studies summary TSS statistics.**

Location	Sample Counts		Frequency Detected (%)	TSS (mg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
Thompson Island (PRW2)	8	0	88	2.48	4.55	7.74	0.68
Schuylerville (Center)	3	0	100	1.55	4.70	7.34	1.69
Waterford High Flow	29	0	100	6.39	127.52	416.00	25.20

*Notes:*

*Statistics based on detectable concentrations only.*

**Table 6-3. Special study summary POC/DOC statistics.**

Location	Sample Counts		Frequency Detected (%)	Organic Carbon (mg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
Dissolved Total Organic Carbon							
Thompson Island (PRW2)	4	0	100	3.98	4.94	6.42	0.58
Schuylerville (Center)	1	0	100	4.56	4.56	4.56	--
Waterford High Flow	29	0	100	2.84	3.80	5.42	0.12
Particulate Organic Carbon							
Thompson Island (PRW2)	4	0	25	0.57	0.57	0.57	--
Schuylerville (Center)	1	0	100	0.64	0.64	0.64	--
Waterford High Flow	29	0	100	0.66	2.16	5.89	0.28

*Notes:*

*Statistics based on detectable concentrations only.*

**Table 6-4. Baseline water additional TSS summary statistics.**

Location	Sample Counts		Frequency Detected (%)	TSS (mg/L)			
	ENV	DUP		Minimum	Average	Maximum	Standard Error
Thompson Island Dam	9	0	89	0.90	3.15	9.18	0.84
Schuylerville (Transect)	9	0	100	1.17	3.23	5.09	0.47

*Notes:*

*Statistics based on detectable concentrations only.*

**Table 8-1. Summary of analytical data quality for 2006 water environmental samples<sup>1</sup>.**

Analysis Fraction	Number of Results Qualified <sup>2</sup>										Total Number of Results <sup>5</sup>	% Completeness <sup>6</sup>	% Unusable Data <sup>7</sup>	% Usable Data <sup>8</sup>	Qualitative Data Quality
	Unqualified Positive Results	U	<J <sup>3</sup>	U*	JN	J	J <sup>4</sup>	UJ	R	UR					
PCB Congeners (Whole Water Extraction) (NE207_03)	5,127	31,198	65	1,519	0	6,070	5,809	128	0	76	44,183	94.8%	0.17%	99.8%	Very Good
Total Metals (200.8)	672	613	NA	310	0	387	380	6	0	0	1,988	79.9%	0.0%	100%	Good
Dissolved Metals (200.8)	602	572	NA	484	0	322	312	6	0	2	1,988	70.0%	0.10%	99.9%	Above Average
Total Mercury (245.1)	0	79	NA	5	0	6	6	0	0	0	90	94.0%	0.0%	100%	Very Good
Dissolved Mercury (245.1)	0	80	NA	7	0	3	3	0	0	0	90	92.0%	0.0%	100%	Very Good
Hardness (130.2)	88	0	NA	0	0	2	NA	0	0	0	90	97.8%	0.0%	100%	Excellent
Total Suspended Solids (160.2)	388	51	NA	0	0	24	NA	0	0	0	463	94.8%	0.0%	100%	Excellent
POC/DTC/DOC (NE128_03)	314	0	NA	213	0	153	NA	1	0	0	681	46.1%	0.0%	100%	Average
ENTIRE WATER SAMPLE DATA SET	7,191	32,593	65	2,538	0	6,967	6,510	141	0	78	49,573	92.5%	0.16%	99.8%	Very Good

*Notes:*

1 - Summary is for water environmental samples and does not include results from Field Duplicates, Field Blanks, Lab Duplicates, Matrix Spikes or Blanks. Summary is based on Qualification of data from verification and validation.

2 - Results are the number of individual analytes in the analysis fraction. For example, there are 113 analytes in the PCB Congener analysis fraction.

3 - Results for Total PCBs where the sum of the positive PCB congener results was greater than 0 but below the sample-specific Total PCB MDL.

4 - Results qualified as estimates due to being below the reporting limit. For example, of the 6,173 NE207\_03 PCB congener (whole water extraction) results that were qualified J, 5,809 results were qualified J due to being below the reporting limit.

5 - Total Number of Results is the summation of all qualified and unqualified results.

6 - The % Completeness is the sum of results that were valid as reported  $[Unqualified\ Positive\ Results + U]/Total\ Number\ of\ Results - <J - J^4$ .

7 - % Unusable Data is the sum of the results qualified R + UR/Total Number of Results.

8 - % Usable Data is the sum of the Unqualified Positive Results + U [ $+<J$  for total PCBs] + U\* + J + JN + UJ/Total Number of Results.

**Table 8-2. Summary of analytical data quality for 2006 fish tissue environmental samples <sup>1</sup>.**

Analysis Fraction	Number of Results Qualified <sup>2</sup>										Total Number of Results <sup>5</sup>	% Completeness <sup>6</sup>	% Unusable Data <sup>7</sup>	% Usable Data <sup>8</sup>	Qualitative Data Quality
	Unqualified Positive Results	U	<J <sup>3</sup>	U*	JN	J	J <sup>4</sup>	UJ	R	UR					
PCBs as Aroclors (NE148_04)	1,418	2,599	NA	0	0	234	145	85	0	0	4,336	95.8%	0.0%	100.0%	Excellent
PCB Congeners (NE013_07)	3,217	1,118	1	201	0	1,623	1,580	0	0	0	6,160	94.7%	0.0%	100.0%	Excellent
ENTIRE FISH TISSUE DATA SET	4,635	3,717	1	201	0	1,857	1,725	85	0	0	10,496	95.2%	0.0%	100.0%	Excellent

*Notes:*

1 - Summary is for fish tissue environmental samples and does not include results from Lab Duplicates, Matrix Spikes or Blanks. Summary is based on Qualification of data from verification and validation.

2 - Results are the number of individual analytes in the analysis fraction. For example, there are 8 analytes in the Total PCBs as Aroclors analysis fraction.

3 - Results for Total PCBs where the sum of the positive PCB congener results was greater than 0 but below the sample-specific Total PCB MDL.

4 - Results qualified as estimates due to being below the reporting limit. For example, of the 234 NE148\_04 results that were qualified J, 145 results were qualified J due to being below the reporting limit.

5 - Total Number of Results is the summation of all qualified and unqualified results.

6 - The % Completeness is the sum of results that were valid as reported  $[Unqualified\ Positive\ Results + U]/Total\ Number\ of\ Results - <J - J^4$ .

7 - % Unusable Data is the sum of the results qualified R + UR/Total Number of Results.

8 - % Usable Data is the sum of the Unqualified Positive Results + U  $[+<J\ for\ total\ PCBs] + U^* + J + JN + UJ/Total\ Number\ of\ Results$ .

**Table 8-3. Summary of water field duplicate results for the modified Green Bay Method in 2006.**

Method	Analyte	Total No. Field Duplicate Pairs	Total No. Field Duplicate Pairs with NDs for Both Samples	Total No. Field Duplicate Pairs with Positives in Either Sample					Overall % Meet Criteria
				Total No.	No. Meet Criteria	No. Do Not Meet Criteria	% Meet Criteria	% Do Not Meet Criteria	
NE207_03	Total PCB	53	8	45	45	0	100	0	100
NE207_03	Peak 2	53	15	38	35	3	92	8	94
NE207_03	Peak 3	53	53	0	0	0	NA	NA	100
NE207_03	Peak 4	53	53	0	0	0	NA	NA	100
NE207_03	Peak 5	53	3	50	48	2	96	4	96
NE207_03	Peak 6	53	42	11	10	1	91	9	98
NE207_03	Peak 7	53	27	26	26	0	100	0	100
NE207_03	Peak 8	53	45	8	8	0	100	0	100
NE207_03	Peak 9	53	53	0	0	0	NA	NA	100
NE207_03	Peak 10	53	5	48	44	4	92	8	92
NE207_03	Peak 11	53	53	0	0	0	NA	NA	100
NE207_03	Peak 12	53	53	0	0	0	NA	NA	100
NE207_03	Peak 13	53	53	0	0	0	NA	NA	100
NE207_03	Peak 14	53	14	39	39	0	100	0	100
NE207_03	Peak 15	53	13	40	39	1	98	3	98
NE207_03	Peak 16	53	10	43	40	3	93	7	94
NE207_03	Peak 17	53	9	44	44	0	100	0	100
NE207_03	Peak 19	53	53	0	0	0	NA	NA	100
NE207_03	Peak 20	53	41	12	9	3	75	25	94
NE207_03	Peak 21	53	12	41	40	1	98	2	98
NE207_03	Peak 22	53	21	32	30	2	94	6	96
NE207_03	Peak 23	53	20	33	32	1	97	3	98
NE207_03	Peak 24	53	27	26	26	0	100	0	100
NE207_03	Peak 25	53	20	33	33	0	100	0	100
NE207_03	Peak 26	53	25	28	28	0	100	0	100

Method	Analyte	Total No. Field Duplicate Pairs	Total No. Field Duplicate Pairs with NDs for Both Samples	Total No. Field Duplicate Pairs with Positives in Either Sample					Overall % Meet Criteria
				Total No.	No. Meet Criteria	No. Do Not Meet Criteria	% Meet Criteria	% Do Not Meet Criteria	
NE207_03	Peak 27	53	36	17	14	3	82	18	94
NE207_03	Peak 28	53	53	0	0	0	NA	NA	100
NE207_03	Peak 29	53	38	15	7	8	47	53	85
NE207_03	Peak 30	53	53	0	0	0	NA	NA	100
NE207_03	Peak 31	53	11	42	42	0	100	0	100
NE207_03	Peak 32	53	6	47	47	0	100	0	100
NE207_03	Peak 33	53	16	37	37	0	100	0	100
NE207_03	Peak 34	53	20	33	33	0	100	0	100
NE207_03	Peak 35	53	52	1	1	0	100	0	100
NE207_03	Peak 36	53	53	0	0	0	NA	NA	100
NE207_03	Peak 37	53	18	35	35	0	100	0	100
NE207_03	Peak 38	53	20	33	33	0	100	0	100
NE207_03	Peak 39	53	18	35	35	0	100	0	100
NE207_03	Peak 41	53	53	0	0	0	NA	NA	100
NE207_03	Peak 42	53	28	25	23	2	92	8	96
NE207_03	Peak 43	53	53	0	0	0	NA	NA	100
NE207_03	Peak 44	53	29	24	21	3	88	13	94
NE207_03	Peak 45	53	48	5	5	0	100	0	100
NE207_03	Peak 46	53	30	23	23	0	100	0	100
NE207_03	Peak 47	53	40	13	13	0	100	0	100
NE207_03	Peak 48	53	31	22	22	0	100	0	100
NE207_03	Peak 49	53	22	31	30	1	97	3	98
NE207_03	Peak 50	53	27	26	26	0	100	0	100
NE207_03	Peak 51	53	19	34	32	2	94	6	96
NE207_03	Peak 52	53	51	2	2	0	100	0	100
NE207_03	Peak 53	53	21	32	32	0	100	0	100

Method	Analyte	Total No. Field Duplicate Pairs	Total No. Field Duplicate Pairs with NDs for Both Samples	Total No. Field Duplicate Pairs with Positives in Either Sample					Overall % Meet Criteria
				Total No.	No. Meet Criteria	No. Do Not Meet Criteria	% Meet Criteria	% Do Not Meet Criteria	
NE207_03	Peak 54	53	25	28	26	2	93	7	96
NE207_03	Peak 55	53	47	6	3	3	50	50	94
NE207_03	Peak 56	53	32	21	12	9	57	43	83
NE207_03	Peak 57	53	26	27	27	0	100	0	100
NE207_03	Peak 58	53	25	28	28	0	100	0	100
NE207_03	Peak 59	53	22	31	30	1	97	3	98
NE207_03	Peak 60	53	43	10	10	0	100	0	100
NE207_03	Peak 61	53	14	39	39	0	100	0	100
NE207_03	Peak 62	53	53	0	0	0	NA	NA	100
NE207_03	Peak 63	53	50	3	2	1	67	33	98
NE207_03	Peak 64	53	45	8	8	0	100	0	100
NE207_03	Peak 65	53	36	17	17	0	100	0	100
NE207_03	Peak 66	53	39	14	7	7	50	50	87
NE207_03	Peak 67	53	34	19	19	0	100	0	100
NE207_03	Peak 68	53	53	0	0	0	NA	NA	100
NE207_03	Peak 69	53	41	12	12	0	100	0	100
NE207_03	Peak 70	53	53	0	0	0	NA	NA	100
NE207_03	Peak 71	53	53	0	0	0	NA	NA	100
NE207_03	Peak 72	53	53	0	0	0	NA	NA	100
NE207_03	Peak 73	53	52	1	1	0	100	0	100
NE207_03	Peak 74	53	39	14	14	0	100	0	100
NE207_03	Peak 75	53	53	0	0	0	NA	NA	100
NE207_03	Peak 76	53	53	0	0	0	NA	NA	100
NE207_03	Peak 77	53	53	0	0	0	NA	NA	100
NE207_03	Peak 78	53	53	0	0	0	NA	NA	100
NE207_03	Peak 79	53	52	1	0	1	0	100	98

Method	Analyte	Total No. Field Duplicate Pairs	Total No. Field Duplicate Pairs with NDs for Both Samples	Total No. Field Duplicate Pairs with Positives in Either Sample					Overall % Meet Criteria
				Total No.	No. Meet Criteria	No. Do Not Meet Criteria	% Meet Criteria	% Do Not Meet Criteria	
NE207_03	Peak 80	53	51	2	2	0	100	0	100
NE207_03	Peak 82	53	47	6	6	0	100	0	100
NE207_03	Peak 83	53	53	0	0	0	NA	NA	100
NE207_03	Peak 84	53	53	0	0	0	NA	NA	100
NE207_03	Peak 85	53	53	0	0	0	NA	NA	100
NE207_03	Peak 87	53	52	1	1	0	100	0	100
NE207_03	Peak 88	53	53	0	0	0	NA	NA	100
NE207_03	Peak 89	53	53	0	0	0	NA	NA	100
NE207_03	Peak 90	53	53	0	0	0	NA	NA	100
NE207_03	Peak 91	53	53	0	0	0	NA	NA	100
NE207_03	Peak 92	53	53	0	0	0	NA	NA	100
NE207_03	Peak 93	53	53	0	0	0	NA	NA	100
NE207_03	Peak 94	53	53	0	0	0	NA	NA	100
NE207_03	Peak 95	53	53	0	0	0	NA	NA	100
NE207_03	Peak 96	53	53	0	0	0	NA	NA	100
NE207_03	Peak 98	53	53	0	0	0	NA	NA	100
NE207_03	Peak 99	53	53	0	0	0	NA	NA	100
NE207_03	Peak 100	53	53	0	0	0	NA	NA	100
NE207_03	Peak 101	53	53	0	0	0	NA	NA	100
NE207_03	Peak 102	53	53	0	0	0	NA	NA	100
NE207_03	Peak 103	53	53	0	0	0	NA	NA	100
NE207_03	Peak 104	53	53	0	0	0	NA	NA	100
NE207_03	Peak 105	53	53	0	0	0	NA	NA	100
NE207_03	Peak 106	53	53	0	0	0	NA	NA	100
NE207_03	Peak 107	53	53	0	0	0	NA	NA	100
NE207_03	Peak 108	53	53	0	0	0	NA	NA	100

Method	Analyte	Total No. Field Duplicate Pairs	Total No. Field Duplicate Pairs with NDs for Both Samples	Total No. Field Duplicate Pairs with Positives in Either Sample					Overall % Meet Criteria
				Total No.	No. Meet Criteria	No. Do Not Meet Criteria	% Meet Criteria	% Do Not Meet Criteria	
NE207_03	Peak 109	53	53	0	0	0	NA	NA	100
NE207_03	Peak 110	53	53	0	0	0	NA	NA	100
NE207_03	Peak 111	53	53	0	0	0	NA	NA	100
NE207_03	Peak 112	53	53	0	0	0	NA	NA	100
NE207_03	Peak 113	53	53	0	0	0	NA	NA	100
NE207_03	Peak 114	53	53	0	0	0	NA	NA	100
NE207_03	Peak 115	53	53	0	0	0	NA	NA	100
NE207_03	Peak 116	53	53	0	0	0	NA	NA	100
NE207_03	Peak 117	53	53	0	0	0	NA	NA	100
NE207_03	Peak 118	53	53	0	0	0	NA	NA	100
NE207_03	All Results <sup>1</sup>	5989	4572	1417	1353	64	95	5	99

Notes:

1 - All Results = Total number Field Duplicate Pairs multiplied by the number of analytes determined by the method.

**Table 8-4. Summary of water field duplicate results for all methods other than the modified Green Bay Method in 2006.**

Method	Analyte	Total No. Field Duplicate Pairs	Total No. Field Duplicate Pairs with NDs for Both Samples	Total No. Field Duplicate Pairs with Positives in Either Sample					Overall % Meet Criteria
				Total No.	No. Meet Criteria	No. Do Not Meet Criteria	% Meet Criteria	% Do Not Meet Criteria	
EPA 200.8	TAL - Aluminum	15	0	15	13	2	87	13	87
EPA 200.8	TAL - Iron	15	0	15	13	2	87	13	87
EPA 200.8	TAL - Lead	15	3	12	12	0	100	0	100
EPA 200.8	TAL - Magnesium	15	0	15	14	1	93	7	93
EPA 200.8	TAL - Manganese	15	0	15	14	1	93	7	93
EPA 200.8	TAL - Nickel	15	1	14	14	0	100	0	100
EPA 200.8	TAL - Potassium	15	1	14	14	0	100	0	100
EPA 200.8	TAL - Silver	15	15	0	0	0	NA	NA	100
EPA 200.8	TAL - Sodium	15	0	15	15	0	100	0	100
EPA 200.8	TAL - Thallium	15	14	1	1	0	100	0	100
EPA 200.8	TAL - Antimony	15	12	3	3	0	100	0	100
EPA 200.8	TAL - Arsenic	15	11	4	4	0	100	0	100
EPA 200.8	TAL - Barium	15	0	15	15	0	100	0	100
EPA 200.8	TAL - Beryllium	15	15	0	0	0	NA	NA	100
EPA 200.8	TAL - Cadmium	15	15	0	0	0	NA	NA	100
EPA 200.8	TAL - Chromium	15	14	1	1	0	100	0	100
EPA 200.8	TAL - Cobalt	15	1	14	14	0	100	0	100
EPA 200.8	TAL - Copper	15	12	3	3	0	100	0	100
EPA 200.8	TAL - Vanadium	15	14	1	0	1	0	100	93
EPA 200.8	TAL - Zinc	15	13	2	2	0	100	0	100
EPA 200.8	TAL - Calcium	15	0	15	15	0	100	0	100
EPA 200.8	TAL - Selenium	15	14	1	1	0	100	0	100
EPA 200.8	All Results <sup>1</sup>	330	155	175	168	7	96	4	98
EPA 200.8	TAL - Aluminum (DISS)	15	3	12	10	2	83	17	87
EPA 200.8	TAL - Iron (DISS)	15	1	14	12	2	86	14	87
EPA 200.8	TAL - Lead (DISS)	15	9	6	6	0	100	0	100
EPA 200.8	TAL - Magnesium (DISS)	15	0	15	14	1	NA	NA	93
EPA 200.8	TAL - Manganese (DISS)	15	0	15	12	3	NA	NA	80
EPA 200.8	TAL - Nickel (DISS)	15	8	7	7	0	100	0	100

Method	Analyte	Total No. Field Duplicate Pairs	Total No. Field Duplicate Pairs with NDs for Both Samples	Total No. Field Duplicate Pairs with Positives in Either Sample					Overall % Meet Criteria
				Total No.	No. Meet Criteria	No. Do Not Meet Criteria	% Meet Criteria	% Do Not Meet Criteria	
EPA 200.8	TAL - Potassium (DISS)	15	2	13	13	0	100	0	100
EPA 200.8	TAL - Silver (DISS)	15	15	0	0	0	NA	NA	100
EPA 200.8	TAL - Sodium (DISS)	15	0	15	15	0	100	0	100
EPA 200.8	TAL - Thallium (DISS)	15	15	0	0	0	NA	NA	100
EPA 200.8	TAL - Antimony (DISS)	15	12	3	3	0	100	0	100
EPA 200.8	TAL - Arsenic (DISS)	15	11	4	4	0	100	0	100
EPA 200.8	TAL - Barium (DISS)	15	0	15	15	0	100	0	100
EPA 200.8	TAL - Beryllium (DISS)	15	15	0	0	0	NA	NA	100
EPA 200.8	TAL - Cadmium (DISS)	15	15	0	0	0	NA	NA	100
EPA 200.8	TAL - Chromium (DISS)	15	15	0	0	0	NA	NA	100
EPA 200.8	TAL - Cobalt (DISS)	15	8	7	3	4	43	57	73
EPA 200.8	TAL - Copper (DISS)	15	15	0	0	0	NA	NA	100
EPA 200.8	TAL - Vanadium (DISS)	15	14	1	1	0	100	0	100
EPA 200.8	TAL - Zinc (DISS)	15	13	2	1	1	50	50	93
EPA 200.8	TAL - Calcium (DISS)	15	0	15	15	0	100	0	100
EPA 200.8	TAL - Selenium (DISS)	15	15	0	0	0	NA	NA	100
EPA 200.8	All Results <sup>1</sup>	330	186	144	131	13	91	9	96
EPA 245.1	TAL - Mercury	15	14	1	1	0	100	0	100
EPA 245.1	TAL - Mercury (DISS)	15	15	0	0	0	NA	NA	100
EPA 130.2	Hardness	15	0	15	13	2	87	13	87
NE128_03	Particulate Organic Carbon	49	32	17	14	3	82	18	94
NE128_03	Dissolved Total Carbon	13	2	11	10	1	91	9	92
NE128_03	Dissolved Total Organic Carbon	47	2	45	43	2	96	4	96
EPA 160.2	Total Suspended Solids	53	6	47	38	9	81	19	83

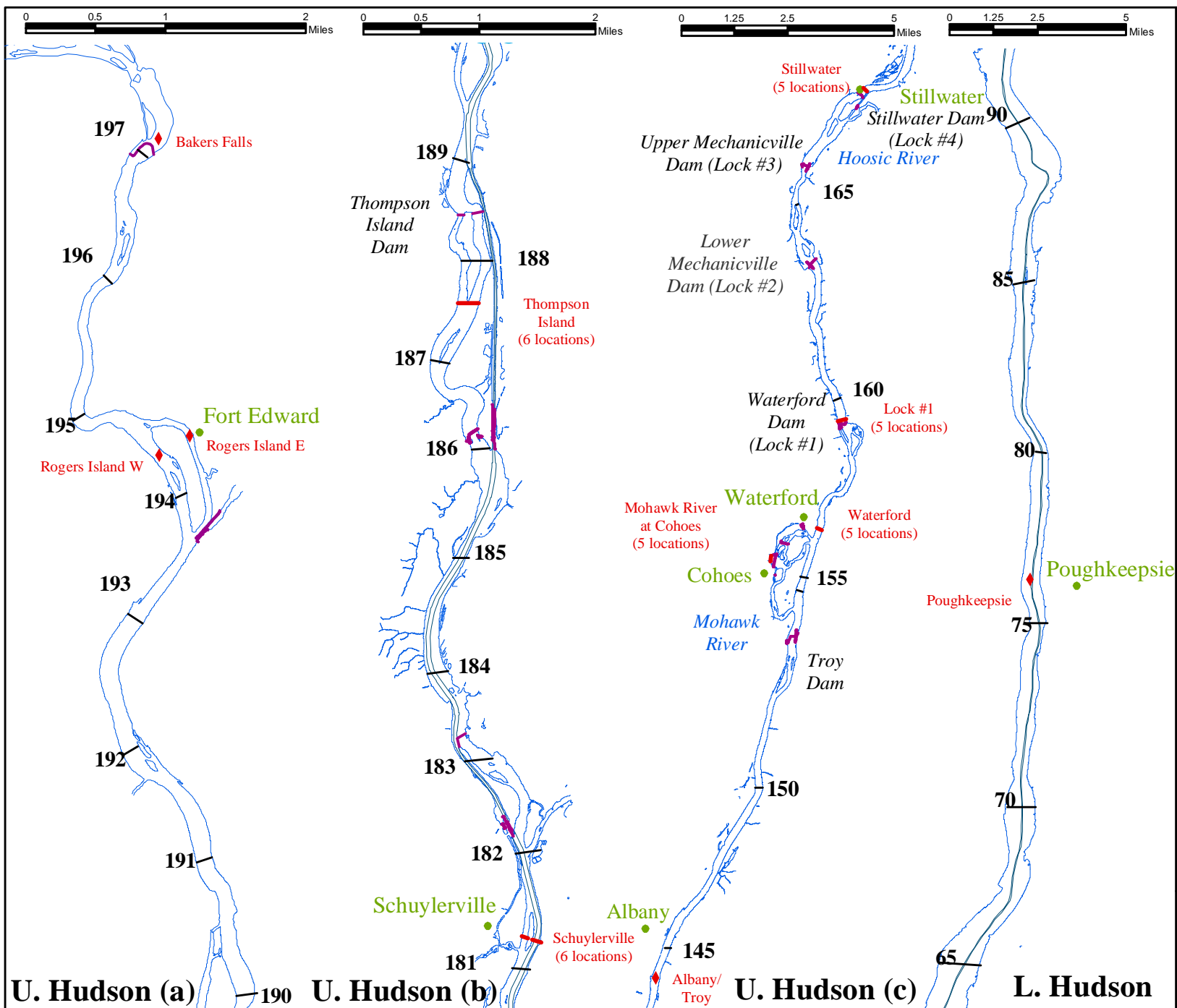
Notes:

1 - All Results = Total number Field Duplicate Pairs multiplied by the number of analytes determined by the method.

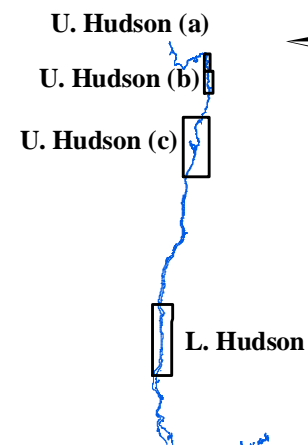
**Table 8-5. Summary statistics of 2006 equipment blanks for water sampling program.**

Analyte	Method	No. Field Blanks	Field Blanks with Results > MDL	Minimum Concentration	Maximum Concentration	Average Concentration	Median Concentration	Concentration Units	% Contaminated
TAL - Aluminum	EPA 200.8	15	3	5.9	10.7	8.3	8.4	ug/L	20%
TAL - Aluminum (DISS)	EPA 200.8	15	4	5.1	11.0	7.0	6.0	ug/L	27%
TAL - Antimony (DISS)	EPA 200.8	15	7	0.045	0.31	0.16	0.16	ug/L	47%
TAL - Arsenic	EPA 200.8	15	2	0.19	0.26	0.23	0.23	ug/L	13%
TAL - Arsenic (DISS)	EPA 200.8	15	1	0.31	0.31	0.31	0.31	ug/L	7%
TAL - Barium	EPA 200.8	15	5	0.11	0.74	0.26	0.16	ug/L	33%
TAL - Barium (DISS)	EPA 200.8	15	5	0.11	0.41	0.20	0.15	ug/L	33%
TAL - Calcium	EPA 200.8	15	11	13.3	74.4	37.8	28.3	ug/L	73%
TAL - Calcium (DISS)	EPA 200.8	15	13	12.1	118	38.4	25.5	ug/L	87%
TAL - Chromium	EPA 200.8	15	14	0.46	2.5	1.2	1.2	ug/L	93%
TAL - Chromium (DISS)	EPA 200.8	15	15	0.28	2.9	1.3	1.1	ug/L	100%
TAL - Cobalt (DISS)	EPA 200.8	15	6	0.030	0.53	0.36	0.40	ug/L	40%
TAL - Copper	EPA 200.8	15	15	0.2	12.1	1.3	0.43	ug/L	100%
TAL - Copper (DISS)	EPA 200.8	15	15	0.22	3.8	0.76	0.61	ug/L	100%
TAL - Iron	EPA 200.8	15	2	4.1	4.7	4.4	4.4	ug/L	13%
TAL - Iron (DISS)	EPA 200.8	15	3	5.6	87.1	34.6	11.0	ug/L	20%
TAL - Lead	EPA 200.8	15	4	0.023	0.088	0.046	0.036	ug/L	27%
TAL - Lead (DISS)	EPA 200.8	15	7	0.033	1.2	0.23	0.079	ug/L	47%
TAL - Magnesium	EPA 200.8	15	2	4.1	4.4	4.3	4.3	ug/L	13%
TAL - Magnesium (DISS)	EPA 200.8	15	4	4.2	6.3	5.2	5.1	ug/L	27%
TAL - Manganese (DISS)	EPA 200.8	15	7	0.1	1.2	0.64	0.66	ug/L	47%
TAL - Nickel	EPA 200.8	15	1	0.24	0.24	0.24	0.24	ug/L	7%
TAL - Nickel (DISS)	EPA 200.8	15	6	0.11	0.40	0.21	0.19	ug/L	40%
TAL - Potassium	EPA 200.8	15	5	15.4	554	127	23.3	ug/L	33%
TAL - Potassium (DISS)	EPA 200.8	15	6	15.8	560	114	23.3	ug/L	40%
TAL - Selenium (DISS)	EPA 200.8	15	2	0.26	0.47	0.37	0.37	ug/L	13%
TAL - Sodium	EPA 200.8	15	10	13.4	342	148	123	ug/L	67%
TAL - Sodium (DISS)	EPA 200.8	15	13	11	287	128	128	ug/L	87%
TAL - Thallium	EPA 200.8	15	2	0.062	0.19	0.13	0.13	ug/L	13%
TAL - Thallium (DISS)	EPA 200.8	15	2	0.059	0.066	0.063	0.063	ug/L	13%
TAL - Vanadium	EPA 200.8	15	4	0.46	1.2	0.84	0.85	ug/L	27%
TAL - Vanadium (DISS)	EPA 200.8	15	5	0.80	17.6	4.4	1.2	ug/L	33%
TAL - Zinc	EPA 200.8	15	14	1.0	14.6	3.1	1.9	ug/L	93%
TAL - Zinc (DISS)	EPA 200.8	15	14	1.4	8.3	3.7	4.1	ug/L	93%
TAL - Mercury	EPA 245.1	15	1	0.066	0.066	0.066	0.066	ug/L	7%
TAL - Mercury (DISS)	EPA 245.1	15	2	0.053	0.099	0.076	0.076	ug/L	13%
DOC	NE128_03	48	1	0.508	0.508	0.508	0.508	mg/L	5%
DTC	NE128_03	13	2	2.24	2.89	2.57	2.57	mg/L	5%
POC	NE128_03	50	45	0.064	0.282	0.128	0.126	mg/L	16%

## FIGURES



## LOCATOR MAP OF THE HUDSON RIVER



### LEGEND

- ♦ Monitoring Stations
- Monitoring Transects
- Navigational Channel
- Dams and Locks
- River Miles

### General Electric Company Hudson River Project

### Figure 2-1. Water Monitoring Stations and Transects.

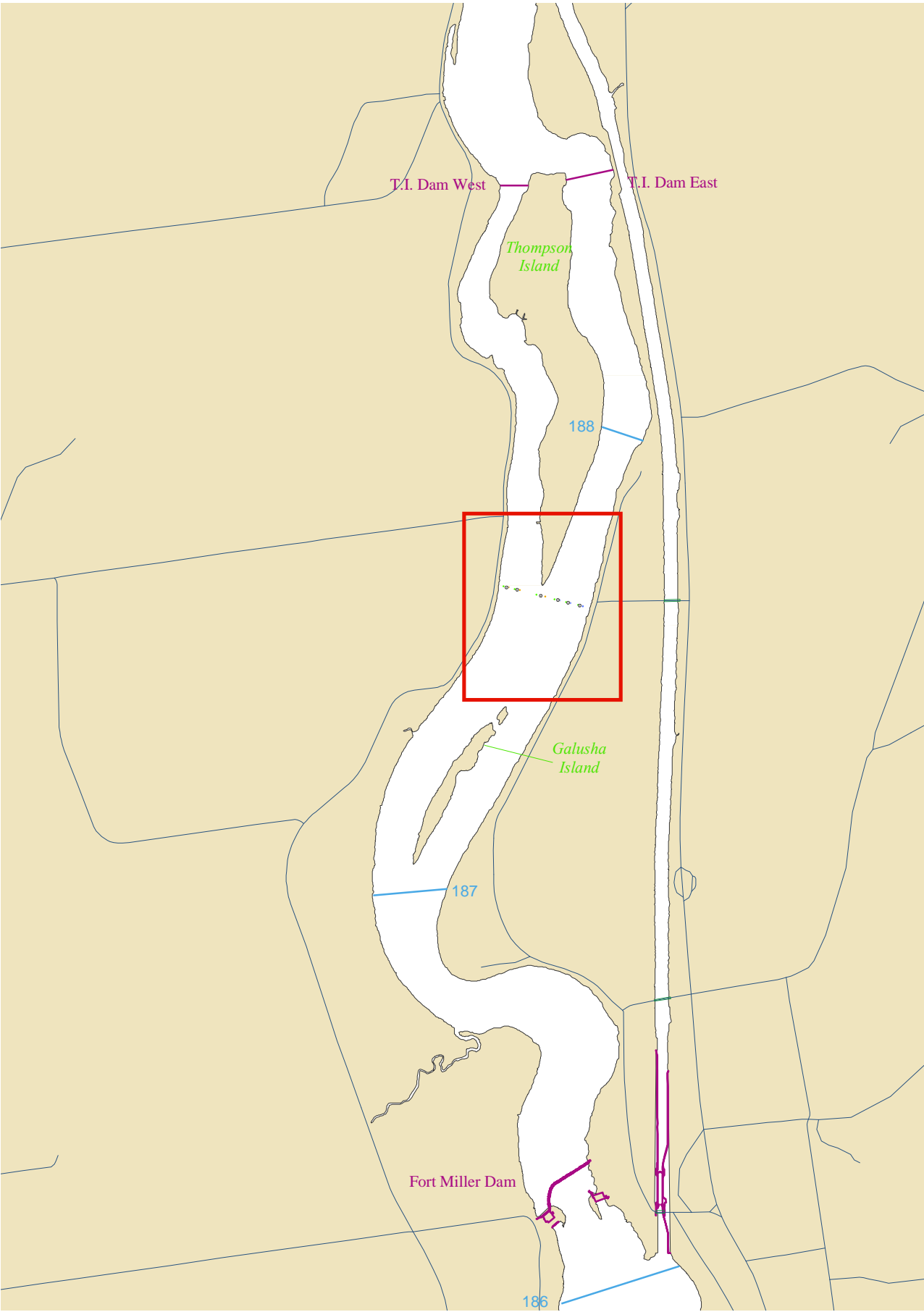
*Note: River miles measured from the Battery (0.0).*



GENbmp:140

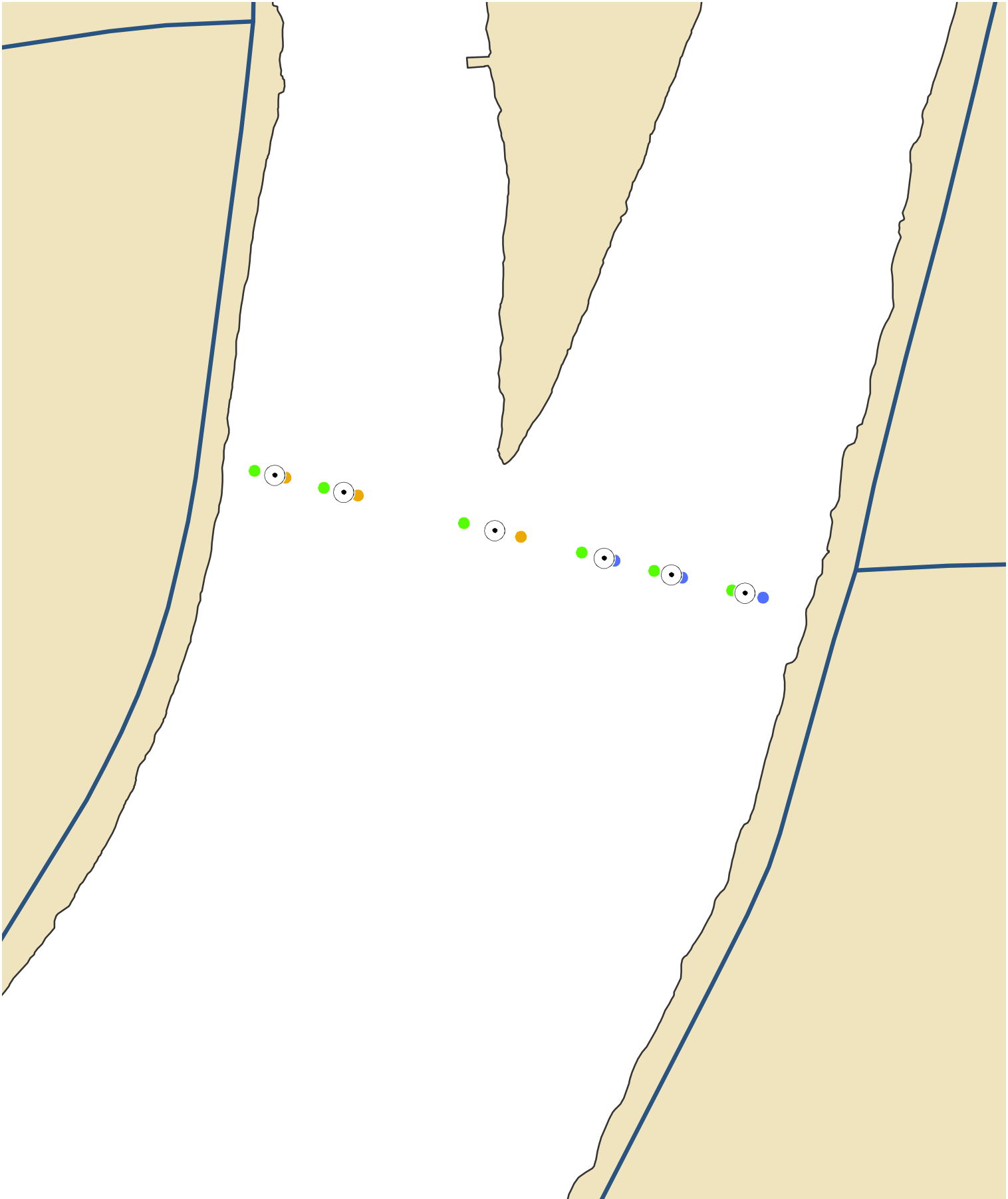
May 2004

OVERVIEW



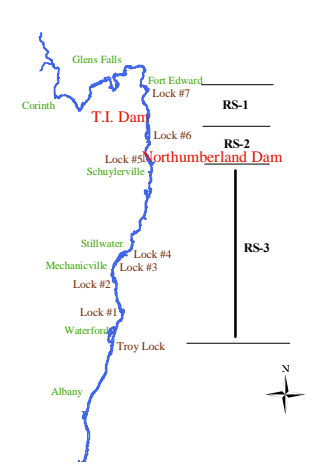
0 2,875 5,750 Feet

FOCUSED AREA



0 150 300 Feet

LOCATOR MAP



Legend

- Sampling Points
- HIGH FLOW
- MODERATE-HIGH FLOW
- MODERATE-LOW FLOW
- LOW FLOW
- bridge
- roads
- River Miles
- Dams and Locks
- Entire\_Hudson\_Polygon
- Land

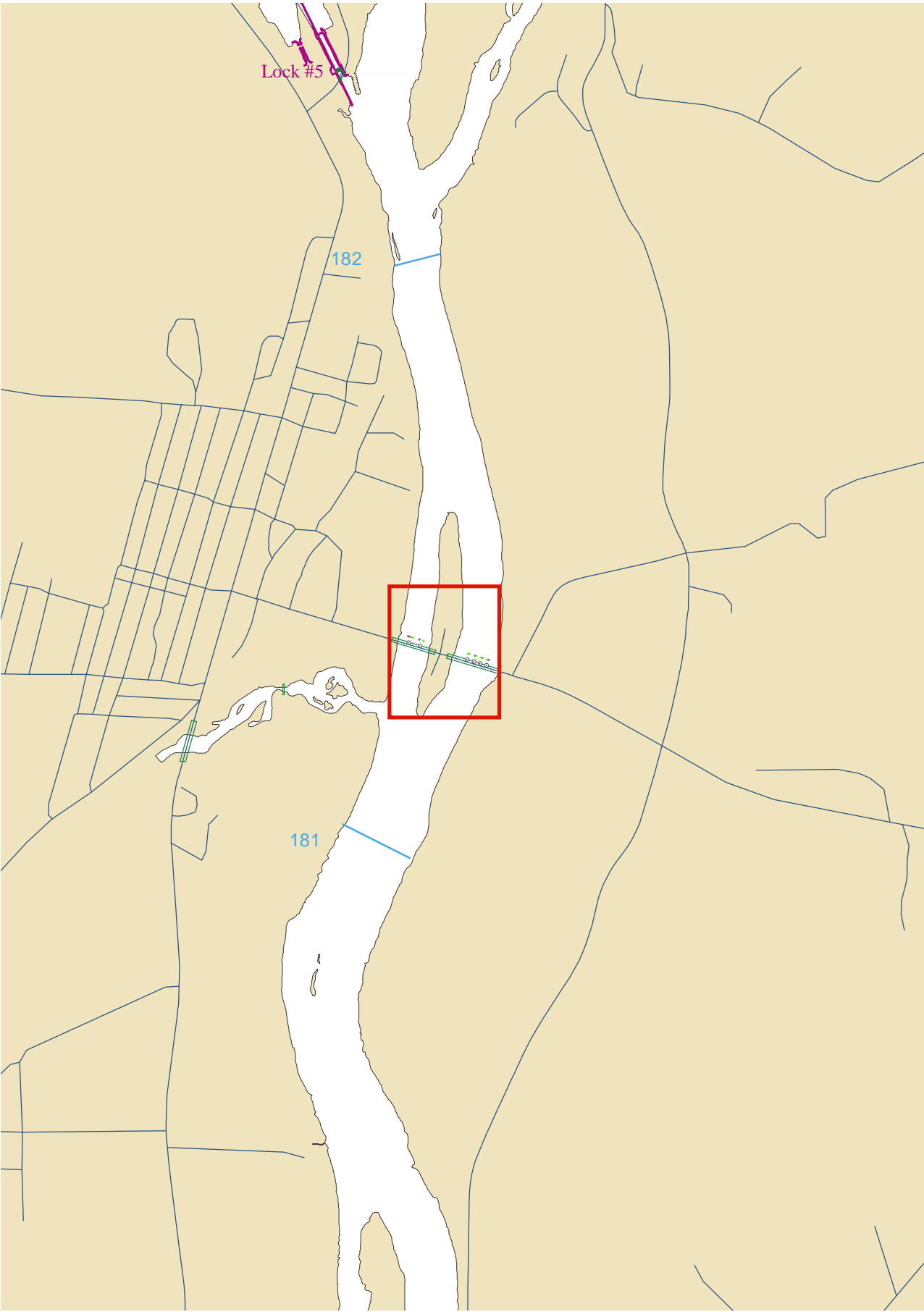
*Note:*  
- Equal discharge locations based on ADCP survey data.  
- Current locations determined by handheld GPS.

General Electric Company  
Hudson River Project

**Figure 2-2**  
**Thompson Island Dam**  
**equal discharge**  
**interval locations**

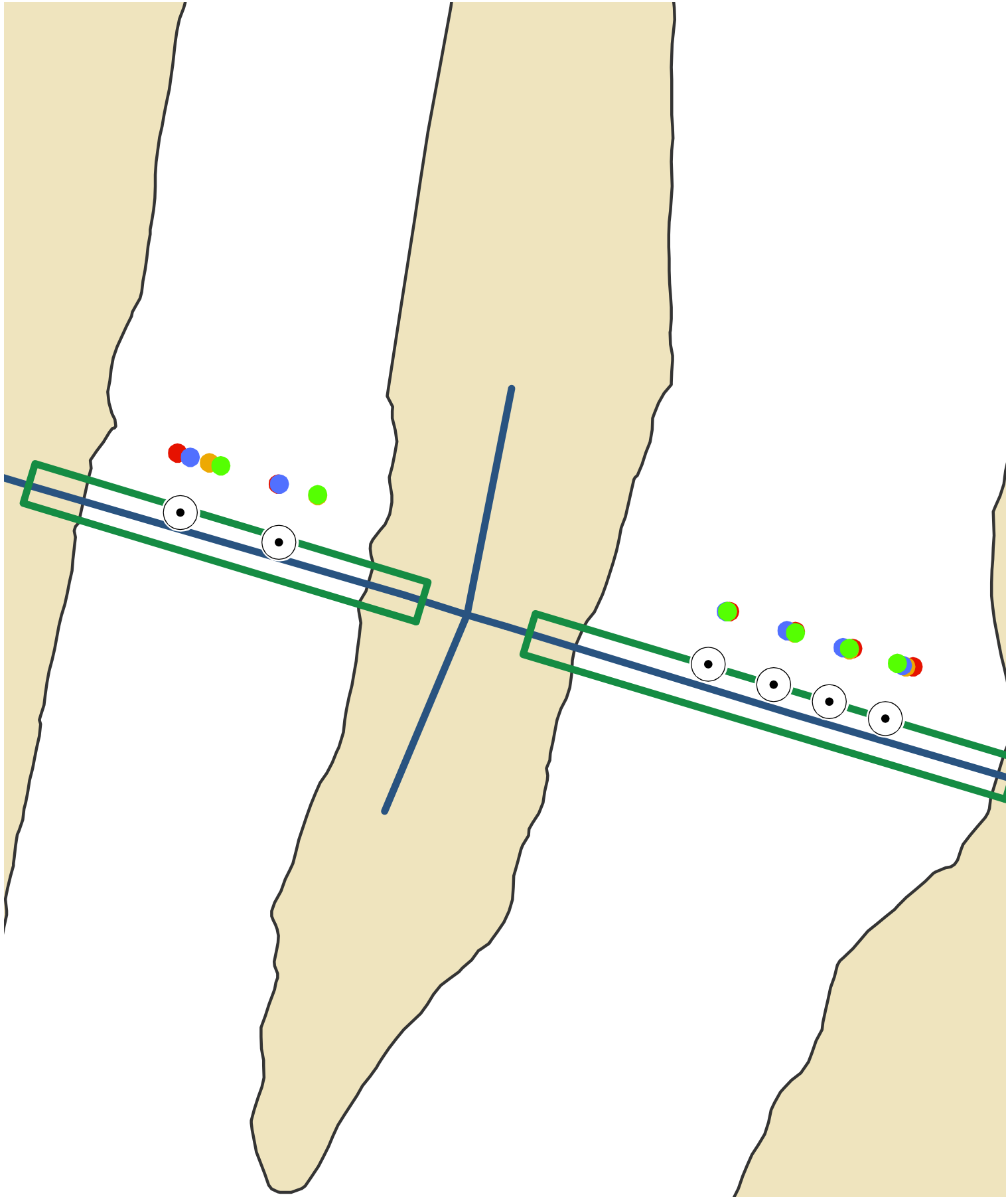


OVERVIEW



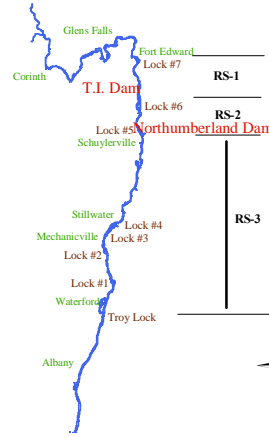
0 2,450 4,900 Feet

FOCUSED AREA



0 150 300 Feet

LOCATOR MAP



Legend

- Sampling Points
- HIGH FLOW
- MODERATE-HIGH FLOW
- MODERATE-LOW FLOW
- LOW FLOW
- bridge
- roads
- River Miles
- Dams and Locks
- Entire\_Hudson\_Polygon
- Land

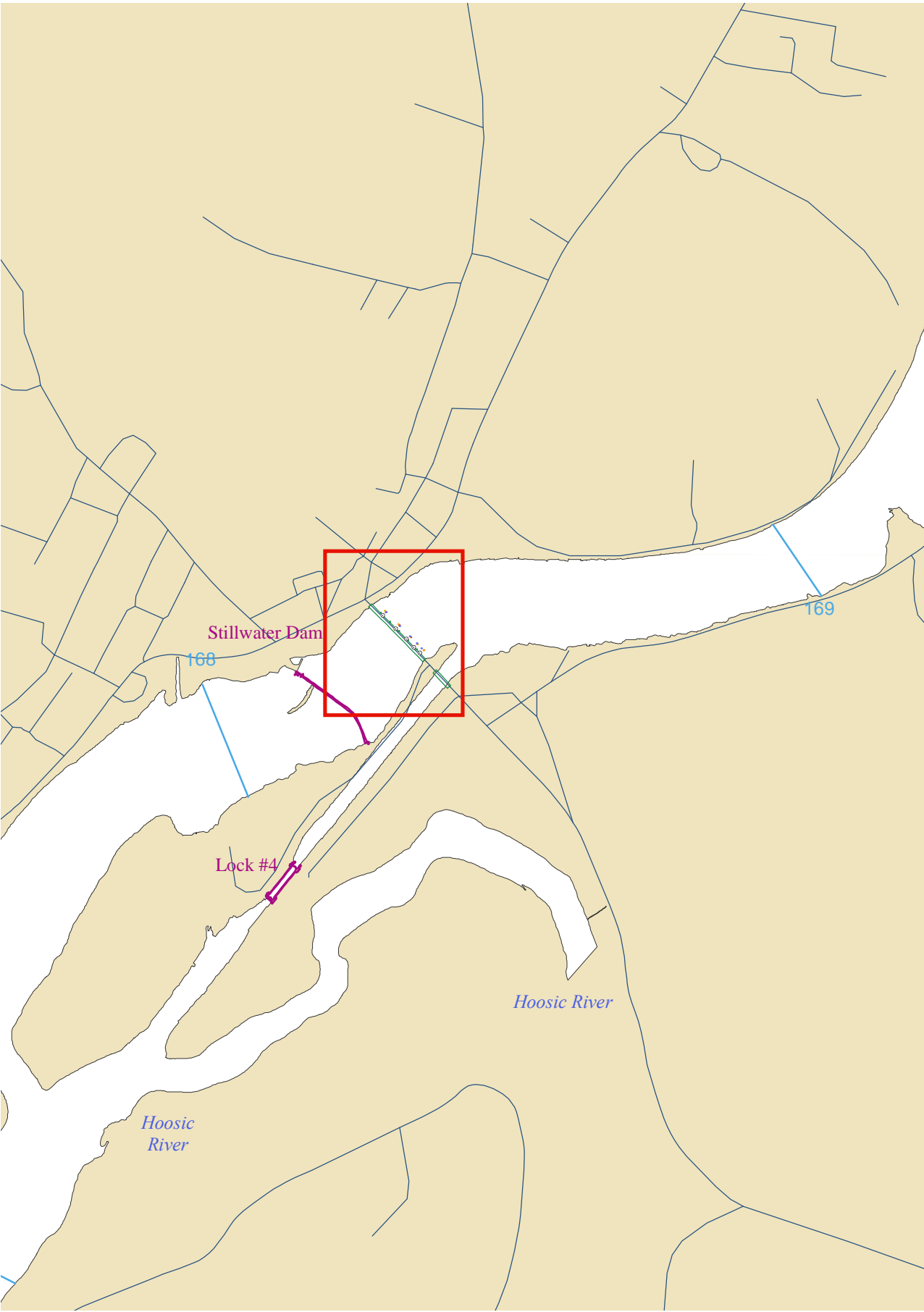
Note:  
- Equal discharge locations based on ADCP survey data.  
- Current locations determined by handheld GPS.

General Electric Company  
Hudson River Project

Figure 2-3  
Schuylerville  
equal discharge  
interval locations

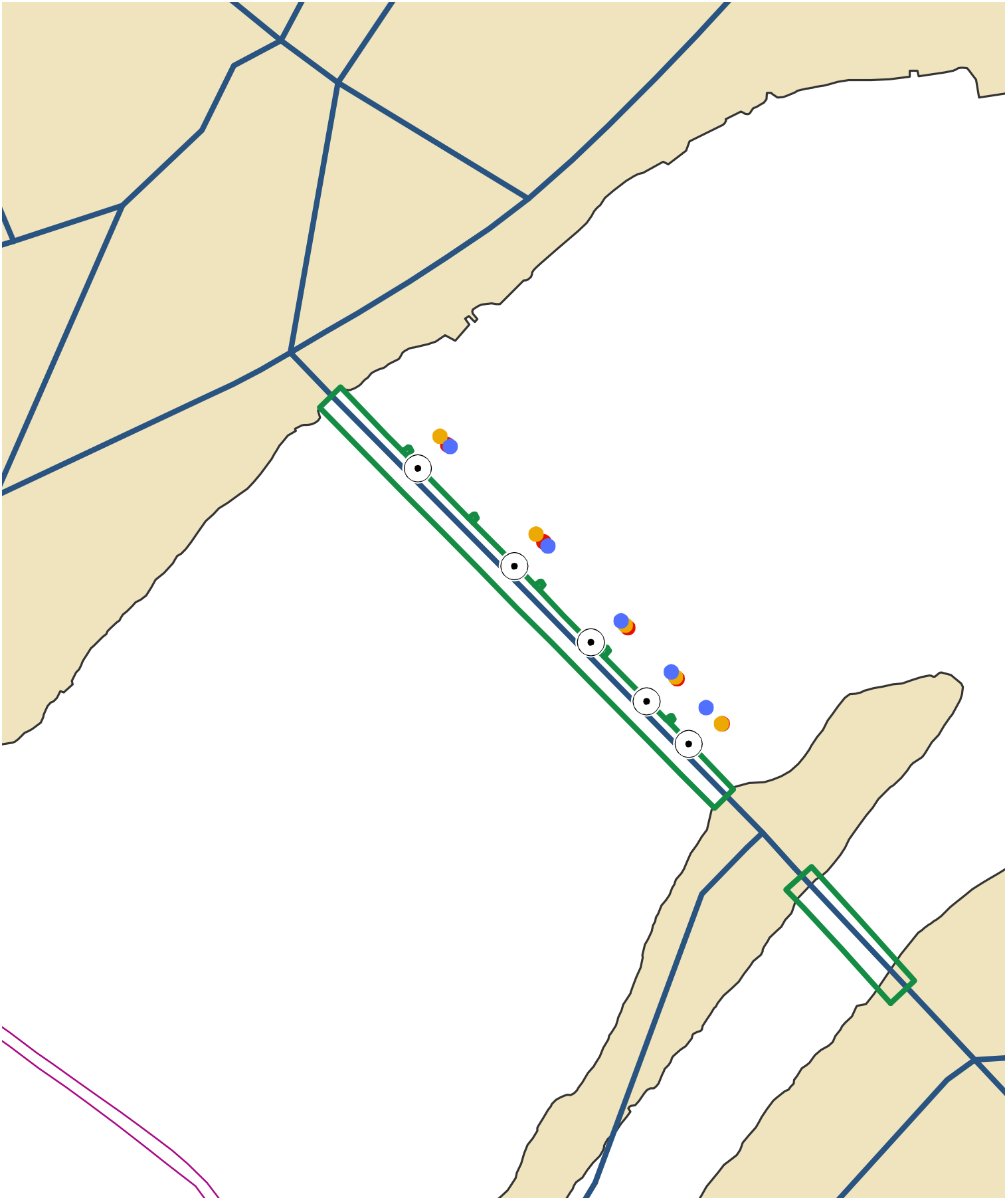


OVERVIEW



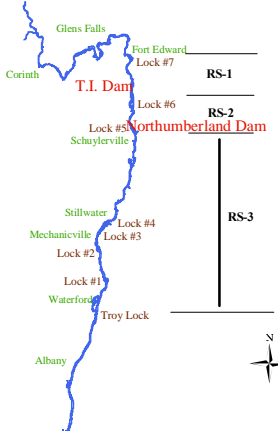
0 2,450 4,900 Feet

FOCUSED AREA



0 150 300 Feet

LOCATOR MAP



Legend

- Sampling Points
- HIGH FLOW
- MODERATE-HIGH FLOW
- MODERATE-LOW FLOW
- LOW FLOW
- bridge
- roads
- River Miles
- Dams and Locks
- Entire\_Hudson\_Polygon
- Land

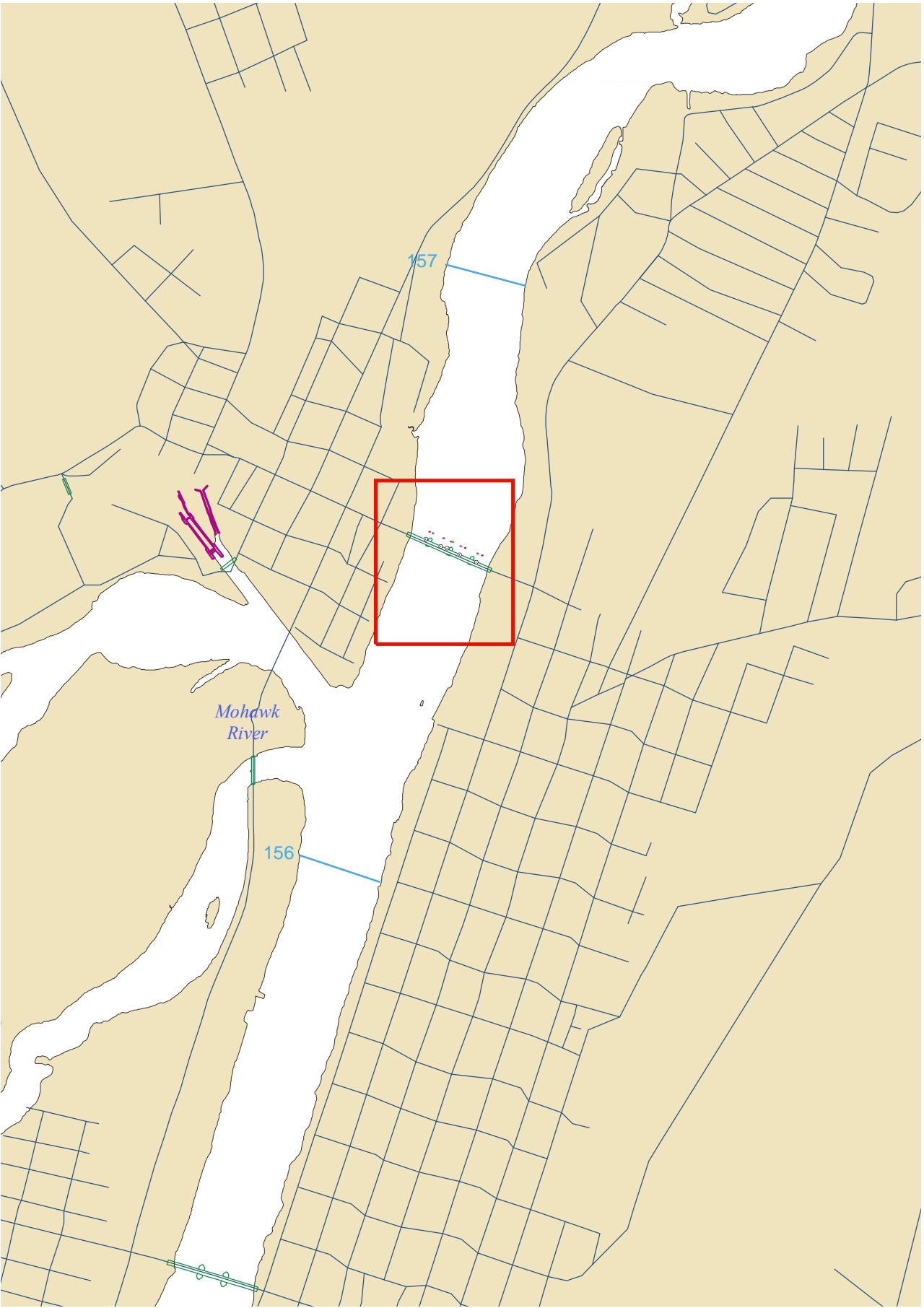
Note:  
- Equal discharge locations based on ADCP survey data.  
- Current locations determined by handheld GPS.

General Electric Company  
Hudson River Project

Figure 2-4  
Stillwater  
equal discharge  
interval locations

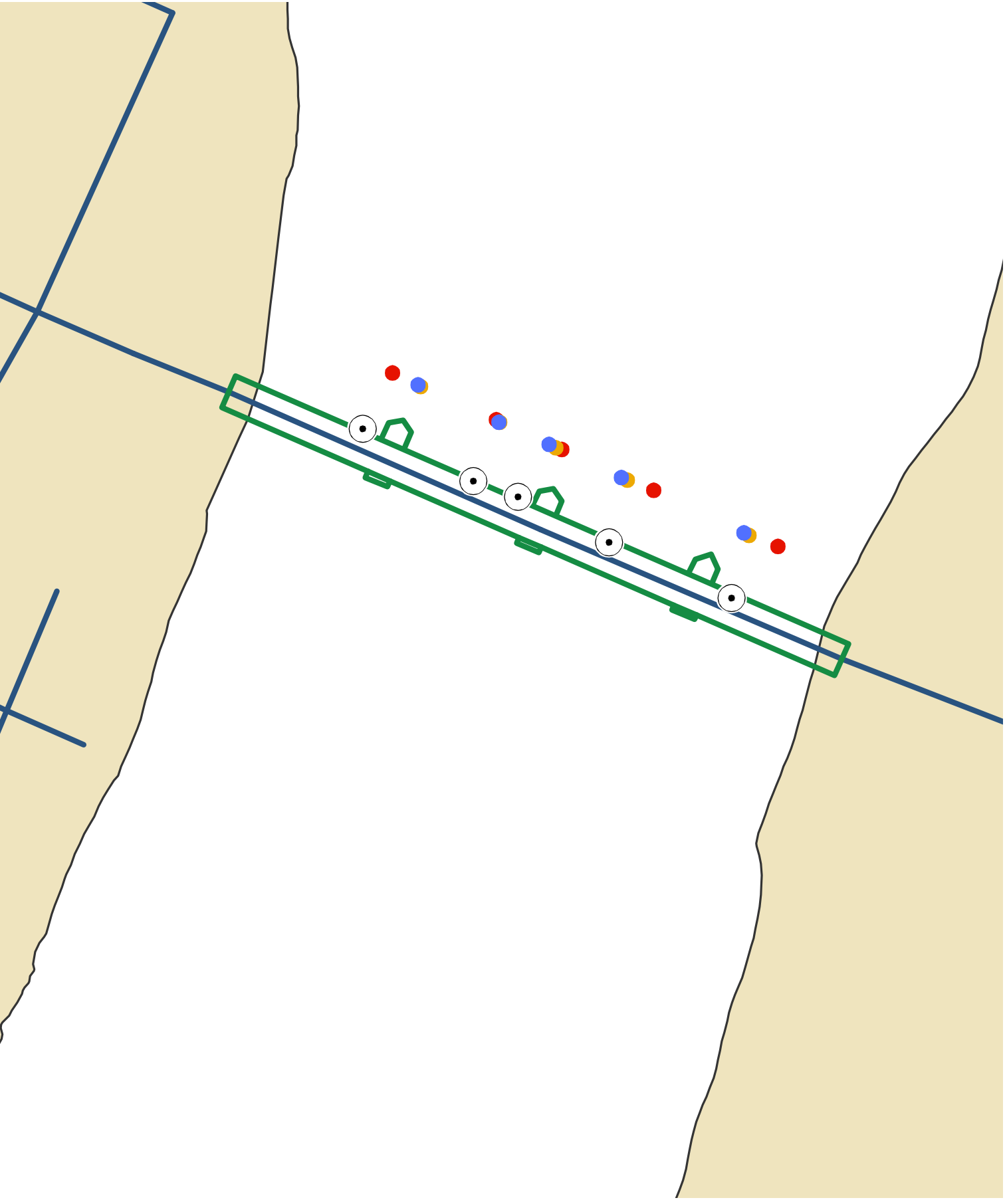


OVERVIEW



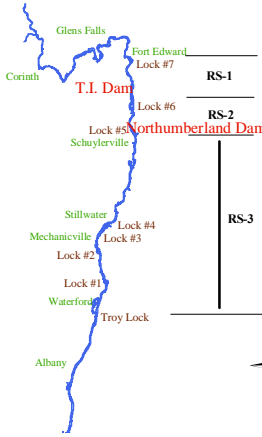
0 2,450 4,900 Feet

FOCUSED AREA



0 150 300 Feet

LOCATOR MAP



Legend

- Sampling Points
- Flow**
  - HIGH FLOW
  - MODERATE-HIGH FLOW
  - MODERATE-LOW FLOW
  - LOW FLOW
- bridge
- roads
- River Miles
- Dams and Locks
- Entire\_Hudson\_Polygon
- Land

*Note:*  
- Equal discharge locations based on ADCP survey data.  
- Current locations determined by handheld GPS.

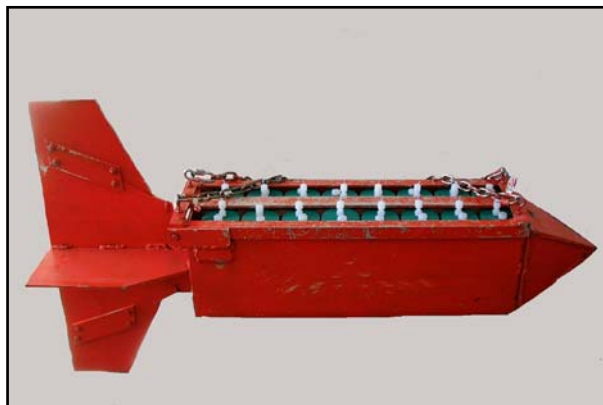
**General Electric Company  
Hudson River Project**

**Figure 2-5  
Waterford  
equal discharge  
interval locations**



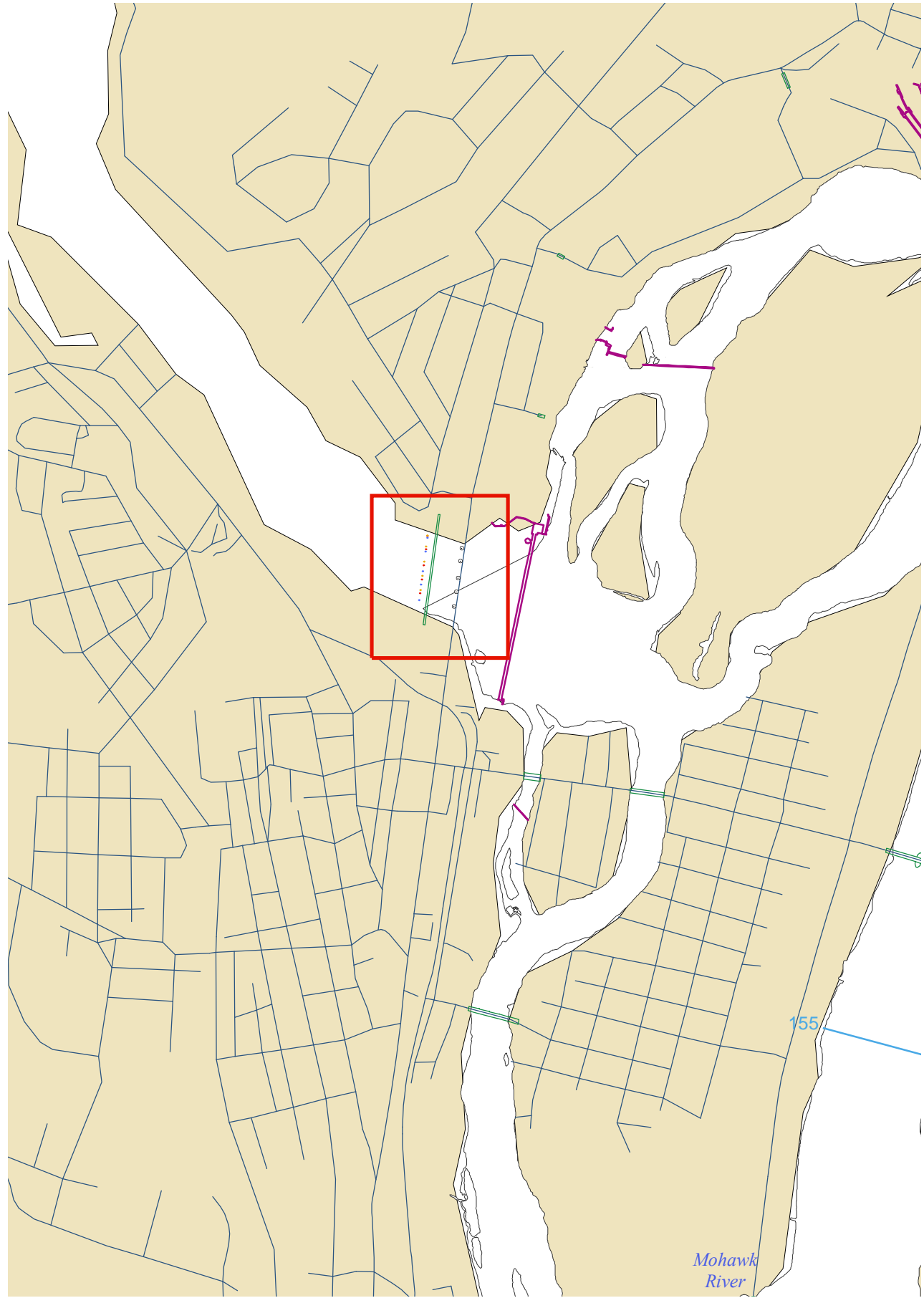


**Figure 2-6. Variable speed bridge and boat cranes for the BMP Water Sampling Program.**



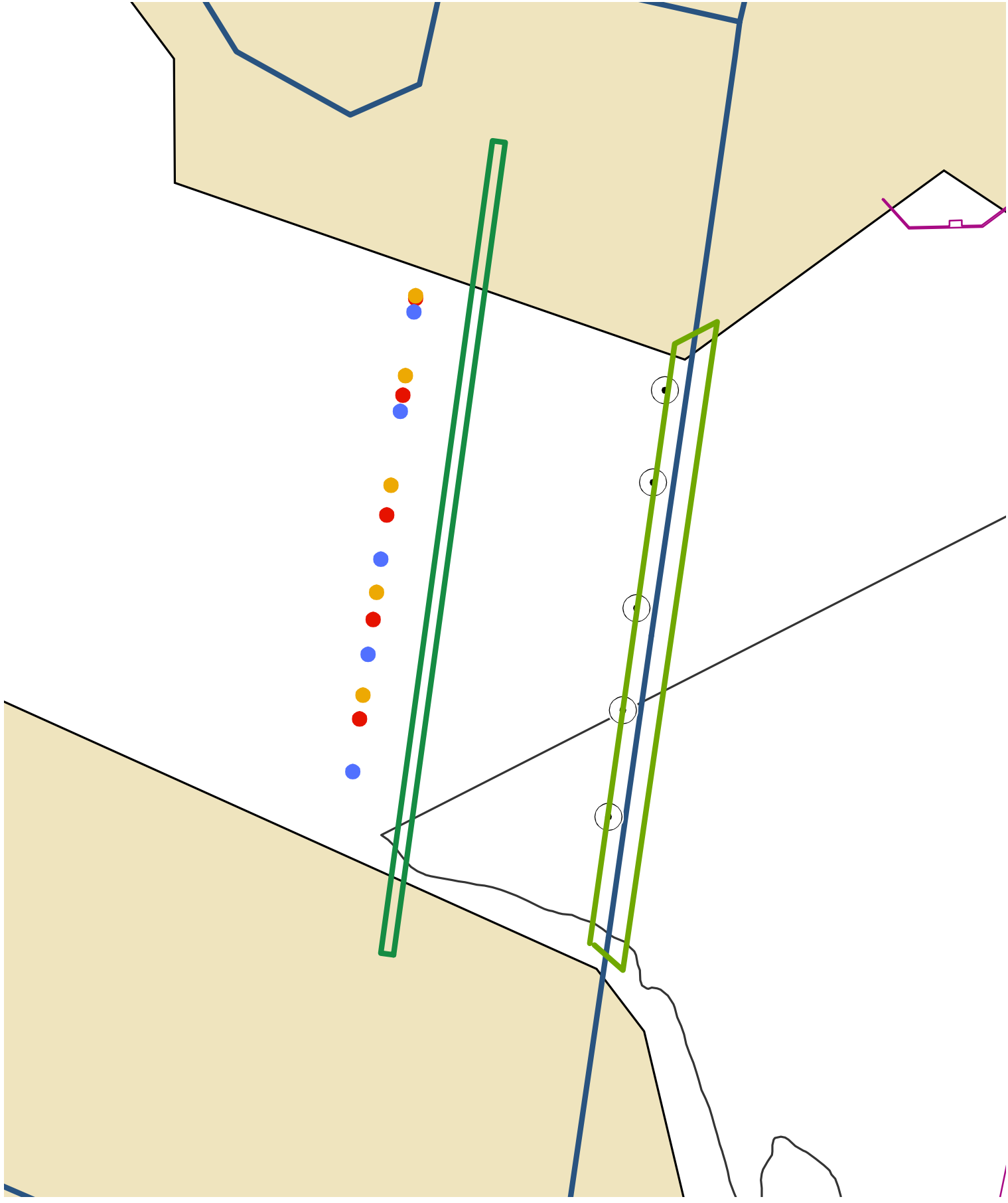
**Figure 2-7. Multiple Aliquot Depth Integrated Sampler (MADIS).**

OVERVIEW



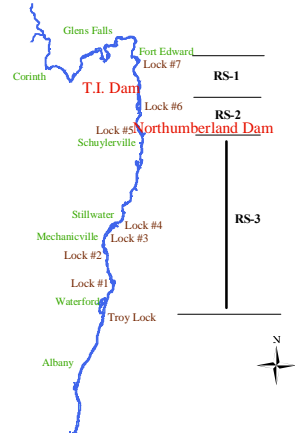
0 2,450 4,900 Feet

FOCUSED AREA



0 150 300 Feet

LOCATOR MAP



Legend

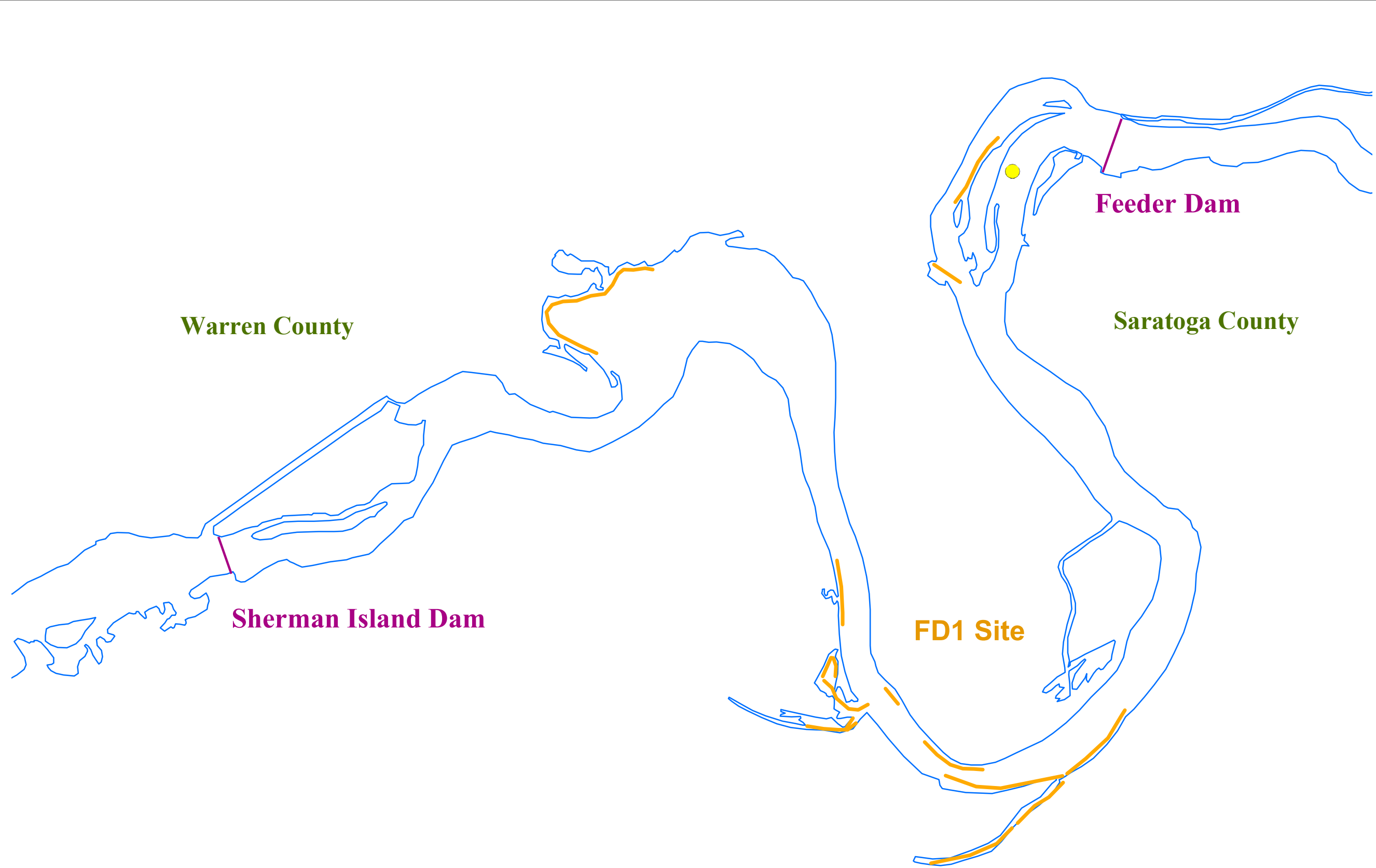
- Sampling Points
- Flow**
  - HIGH FLOW
  - MODERATE-HIGH FLOW
  - MODERATE-LOW FLOW
  - LOW FLOW
- bridge
- roads
- River Miles
- Dams and Locks
- Entire\_Hudson\_Polygon
- Land

*Note:*  
- Equal discharge locations based on ADCP survey data.  
- Current locations determined by handheld GPS.

**General Electric Company  
Hudson River Project**

**Figure 2-8  
Mohawk  
equal discharge  
interval locations**





**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

Glens Falls  
Corinth  
Schuylerville  
Stillwater  
Mechanicville  
Waterford  
Albany  
Schoharie Center

RS-1  
RS-2  
RS-3

N

**GRAPHIC SCALE**

0 0.25 0.5 Miles

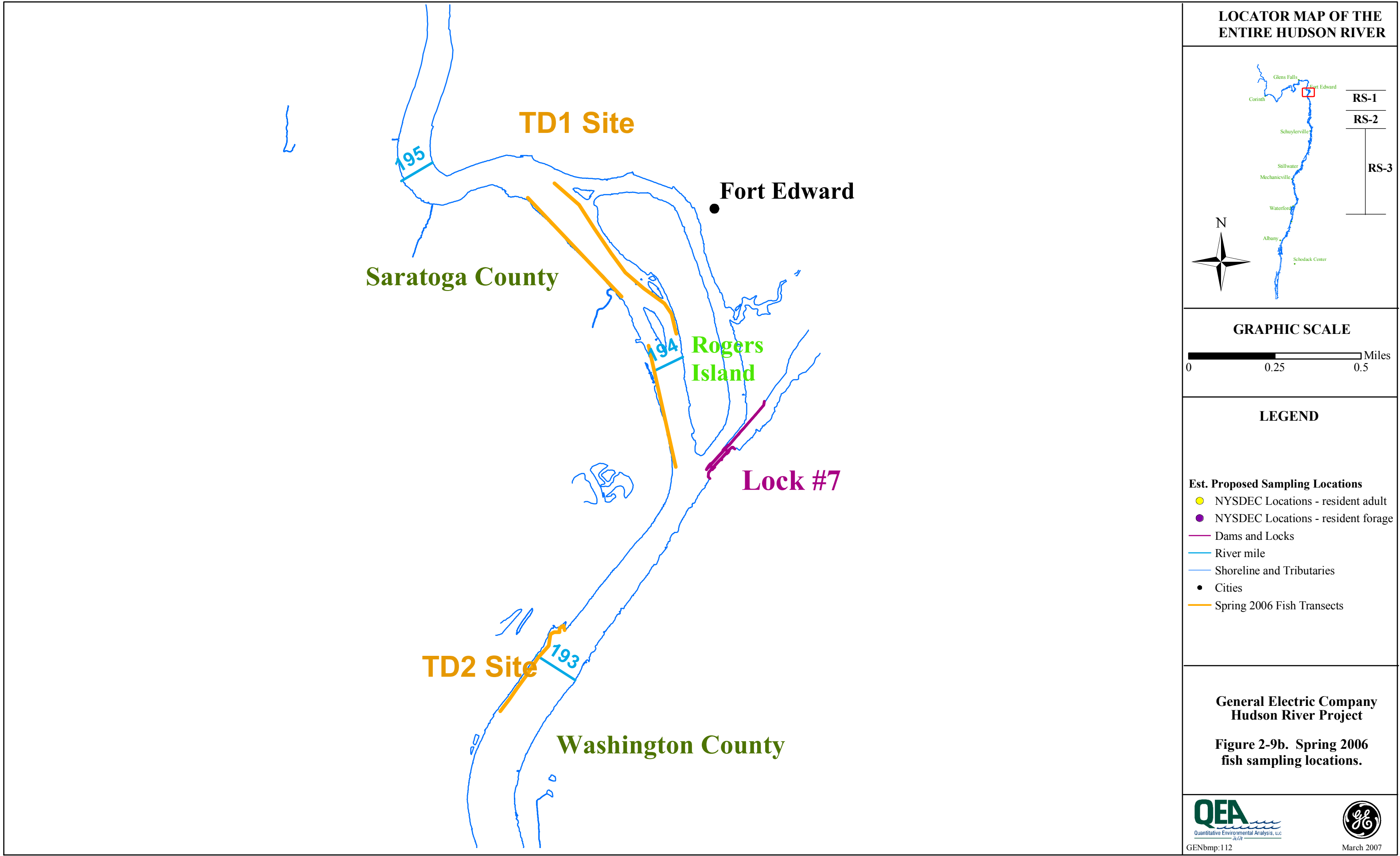
**LEGEND**

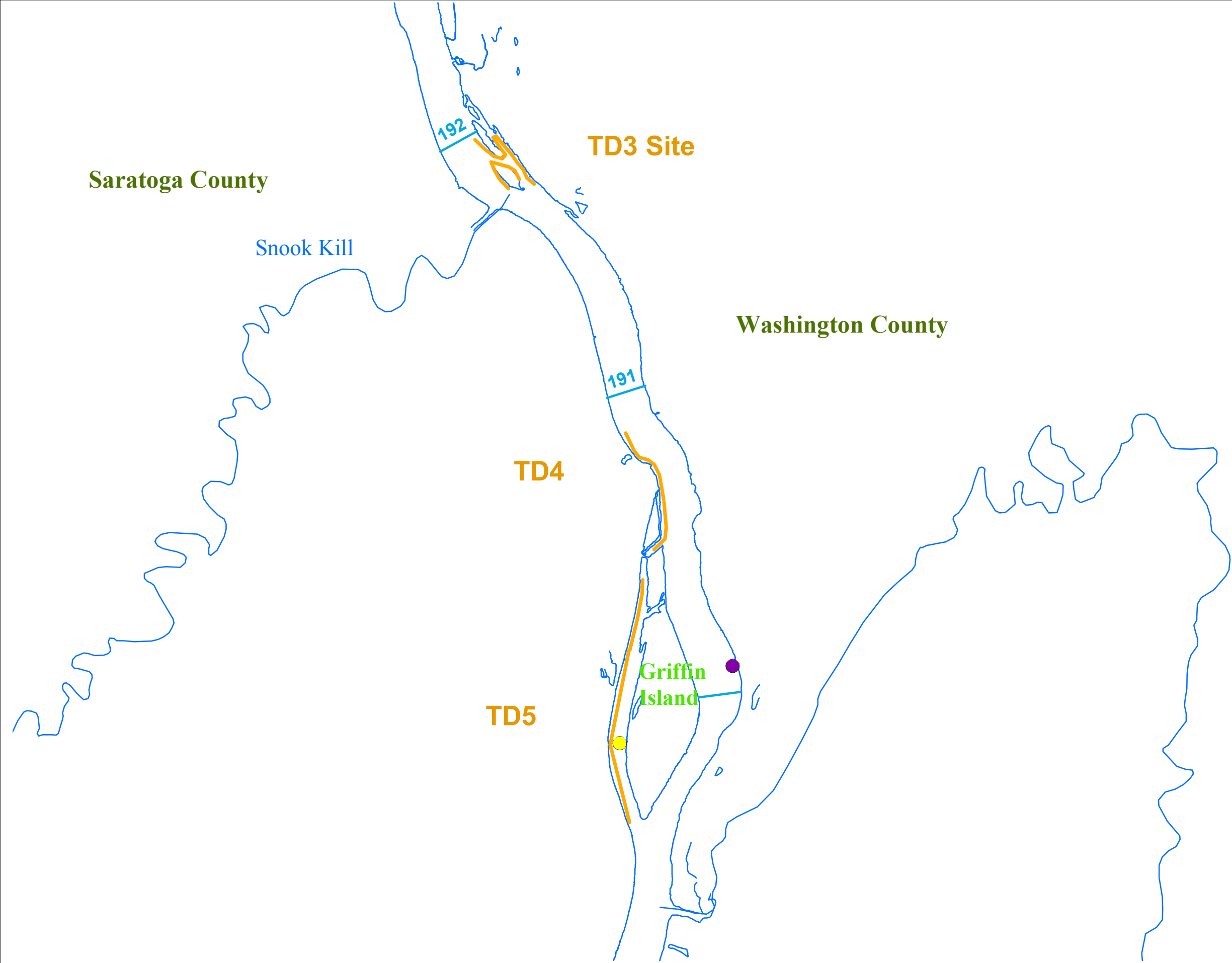
- Est. Proposed Sampling Locations
  - NYSDEC Locations - resident adult (yellow dot)
  - NYSDEC Locations - resident forage (purple dot)
- Dams and Locks (purple line)
- River mile (blue line)
- Shoreline and Tributaries (light blue line)
- Cities (black dot)
- Spring 2006 Fish Transects (orange line)

**General Electric Company  
Hudson River Project**

**Figure 2-9a Spring 2006  
fish sampling locations.**

GENbmp:112 March 2007





**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

**GRAPHIC SCALE**

**LEGEND**  

**Est. Proposed Sampling Locations**

- NYSDEC Locations - resident adult
- NYSDEC Locations - resident forage
- Dams and Locks
- River mile
- Shoreline and Tributaries
- Cities
- Spring 2006 Fish Transects

**General Electric Company  
Hudson River Project**

**Figure 2-9c. Spring 2006  
fish sampling locations.**

GENbmp:112

March 2007

Saratoga County

Washington County

ND1 Site

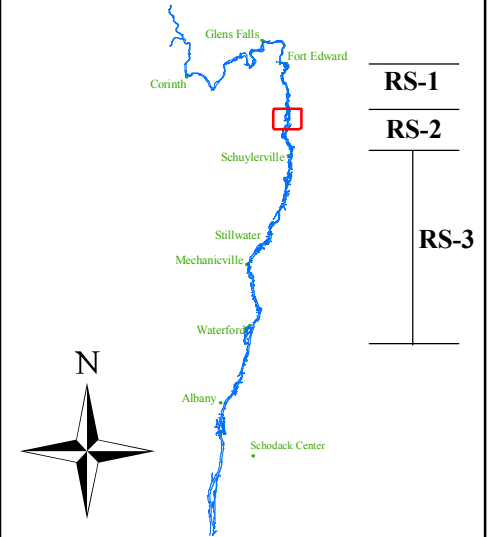
ND2 Site

Fort Miller Dam

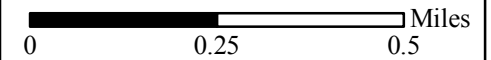
Lock #6

ND3 Site

LOCATOR MAP OF THE  
ENTIRE HUDSON RIVER



GRAPHIC SCALE



LEGEND

Est. Proposed Sampling Locations

- NYSDEC Locations - resident adult
- NYSDEC Locations - resident forage
- Dams and Locks
- River mile
- Shoreline and Tributaries
- Cities
- Spring 2006 Fish Transects

General Electric Company  
Hudson River Project

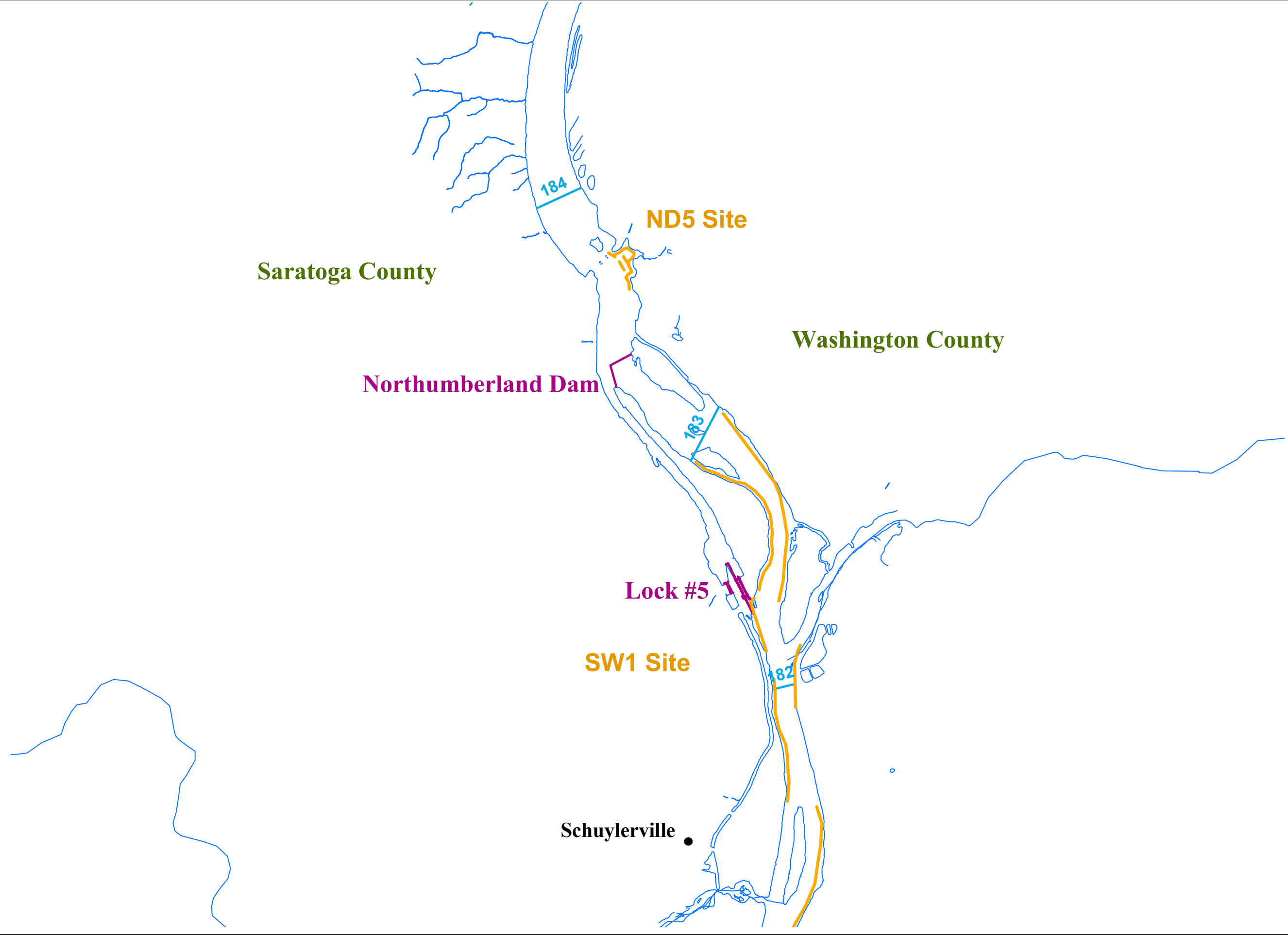
Figure 2-9d. Spring 2006  
fish sampling locations.



GENbmp:112



March 2007



**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

Glens Falls  
Fort Edward  
Corinth  
Schuylerville  
Stillwater  
Mechanicville  
Waterford  
Albany  
Schoharie Center

RS-1  
RS-2  
RS-3

N

**GRAPHIC SCALE**

0 0.25 0.5 Miles

**LEGEND**

**Est. Proposed Sampling Locations**

- NYSDEC Locations - resident adult
- NYSDEC Locations - resident forage
- Dams and Locks
- River mile
- Shoreline and Tributaries
- Cities
- Spring 2006 Fish Transects

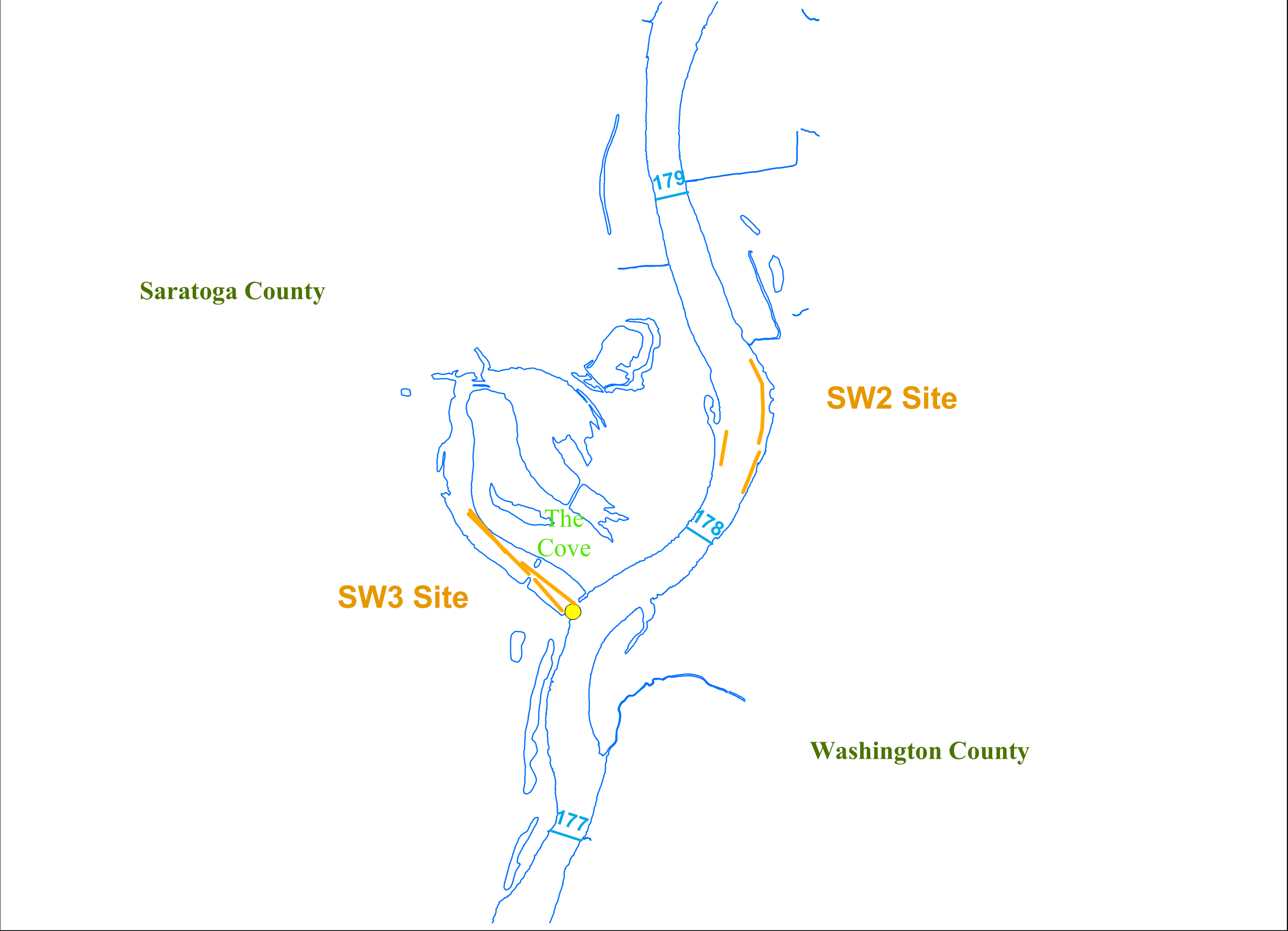
**General Electric Company  
Hudson River Project**

**Figure 2-9e. Spring 2006  
fish sampling locations.**

**QEA**  
Quantitative Environmental Analysis, LLC  
2007

March 2007

GENbmp:112



**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

**GRAPHIC SCALE**

0 0.25 0.5 Miles

**LEGEND**

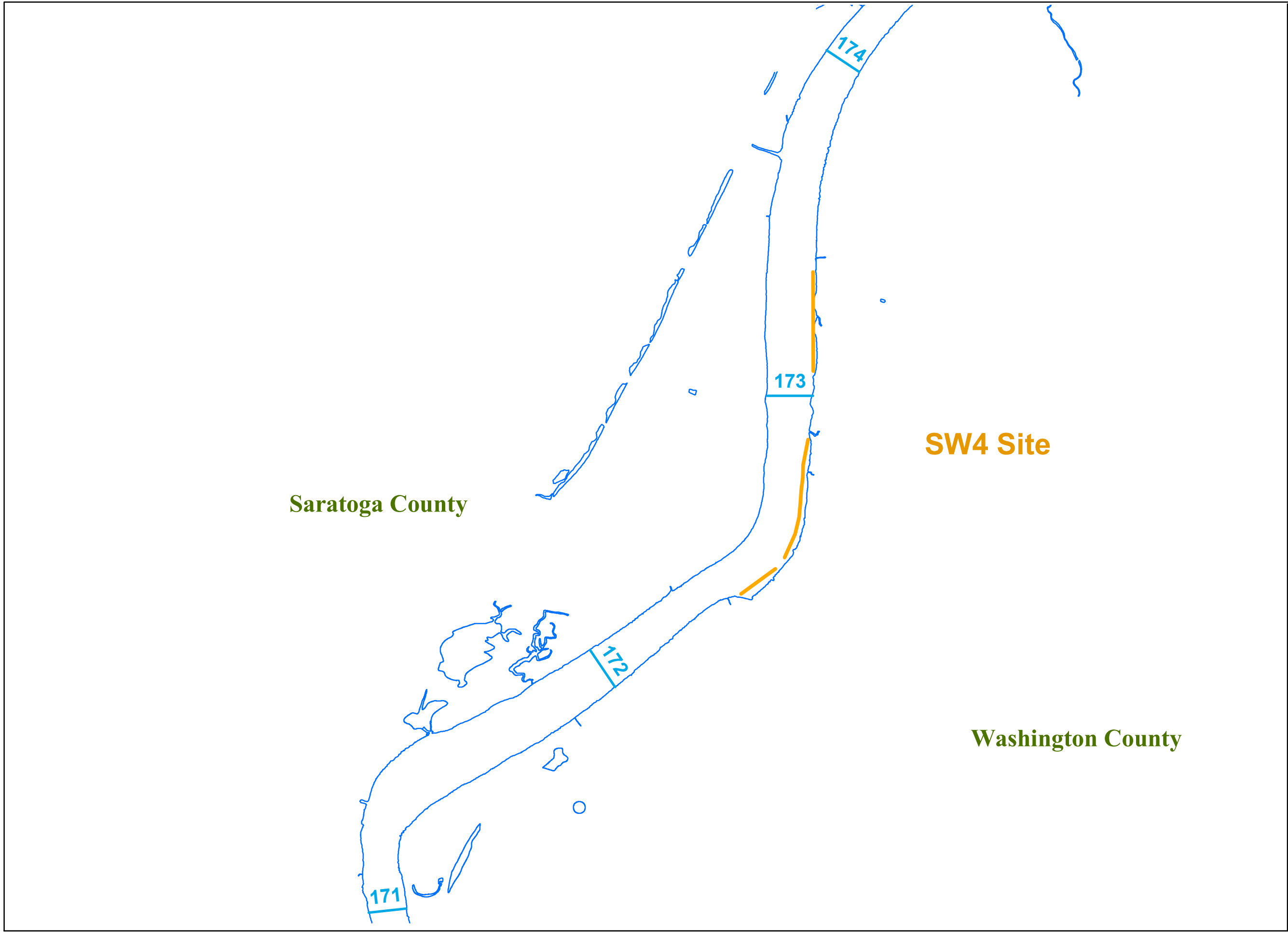
**Est. Proposed Sampling Locations**

- NYSDEC Locations - resident adult
- NYSDEC Locations - resident forage
- Dams and Locks
- River mile
- Shoreline and Tributaries
- Cities
- Spring 2006 Fish Transects

**General Electric Company  
Hudson River Project**

**Figure 2-9f. Spring 2006  
fish sampling locations.**

GENbmp:112 March 2007



**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

**GRAPHIC SCALE**  
Miles  
0 0.25 0.5

**LEGEND**

**Est. Proposed Sampling Locations**

- NYSDEC Locations - resident adult
- NYSDEC Locations - resident forage
- Dams and Locks
- River mile
- Shoreline and Tributaries
- Cities
- Spring 2006 Fish Transects

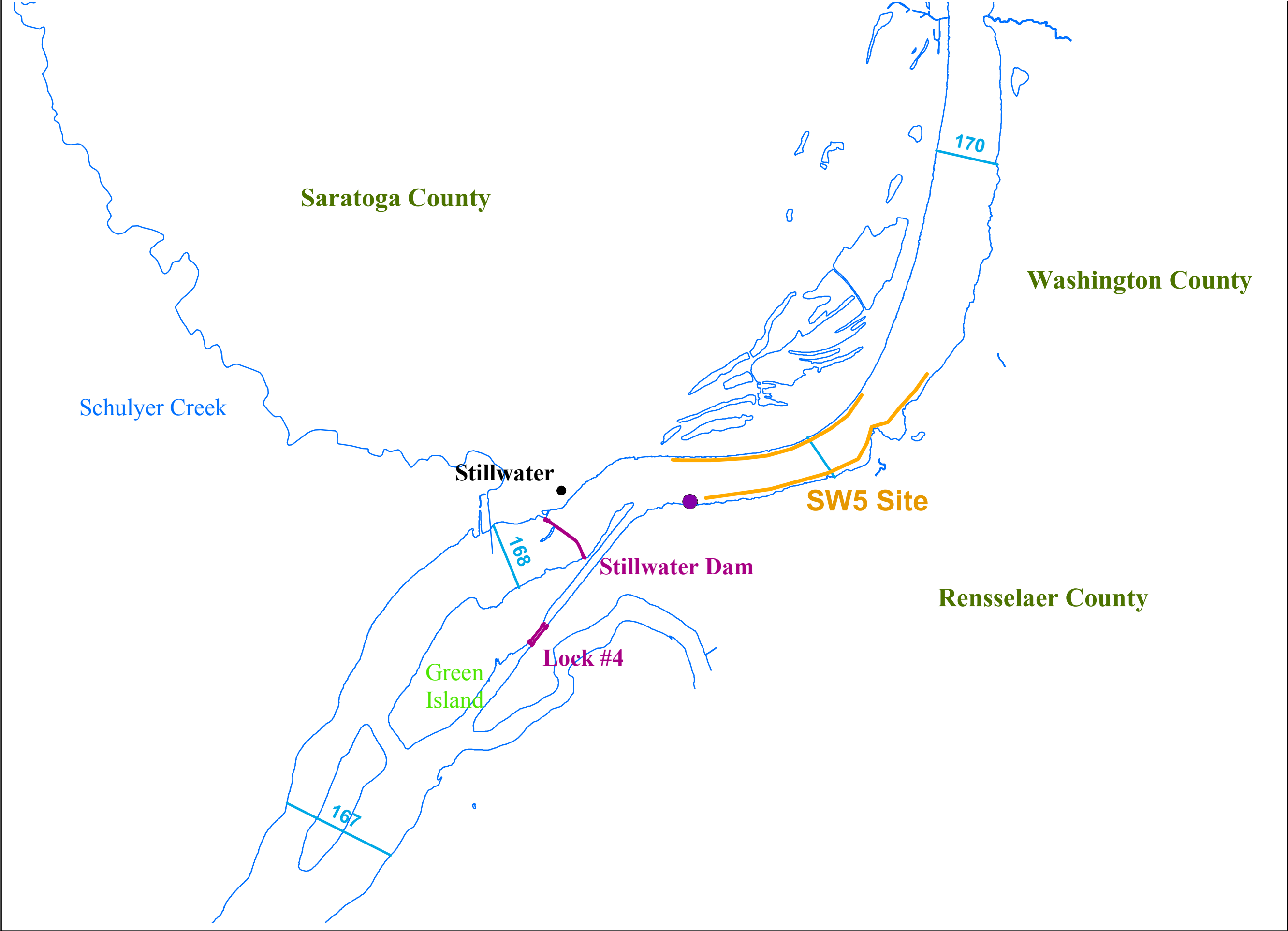
**General Electric Company  
Hudson River Project**

**Figure 2-9g Spring 2006  
fish sampling locations.**

QEA  
Quantitative Environmental Analysis, LLC

March 2007

GENbmp:112



**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

N

**GRAPHIC SCALE**

0 0.25 0.5 Miles

**LEGEND**

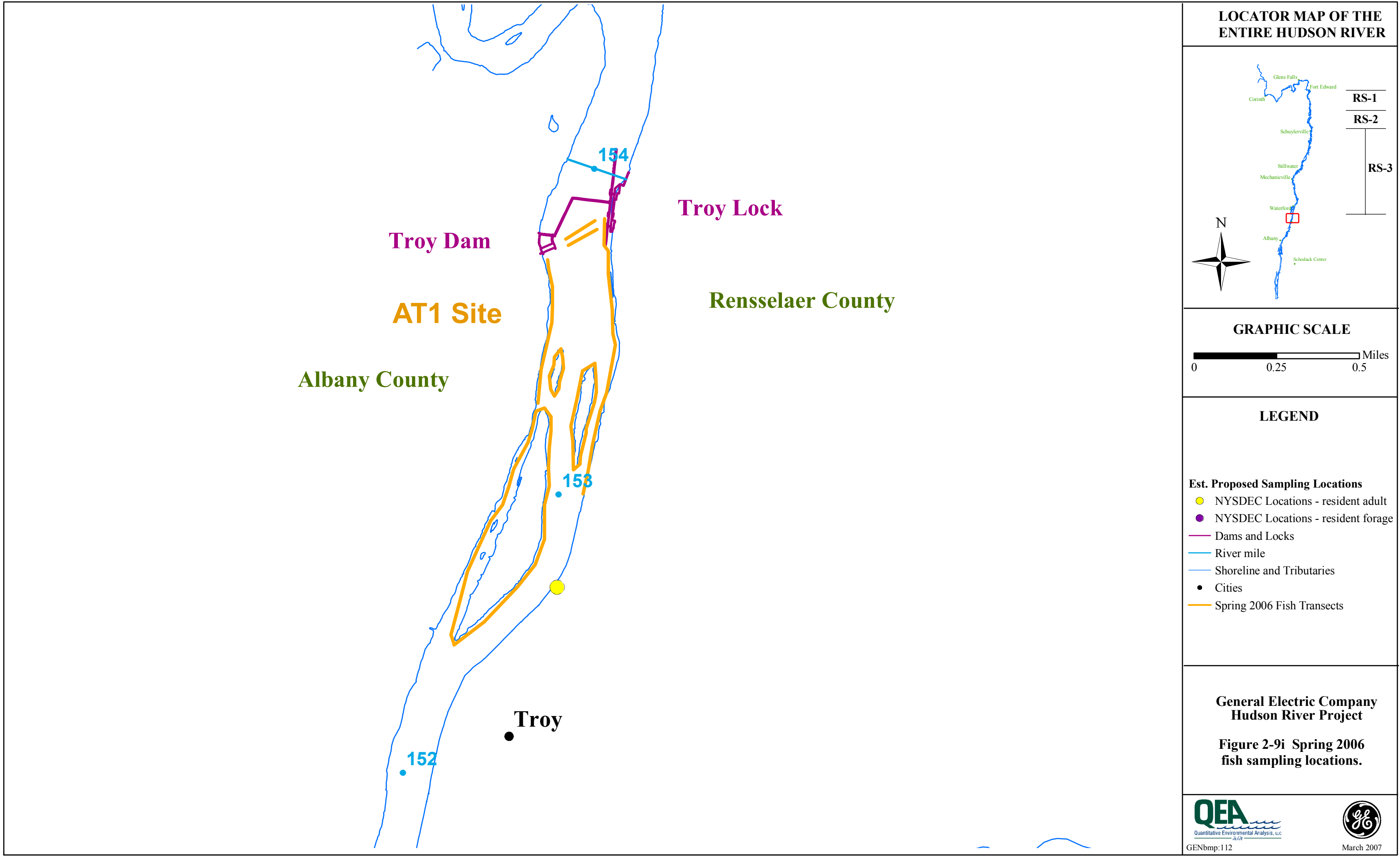
**Est. Proposed Sampling Locations**

- NYSDEC Locations - resident adult
- NYSDEC Locations - resident forage
- Dams and Locks
- River mile
- Shoreline and Tributaries
- Cities
- Spring 2006 Fish Transects

**General Electric Company  
Hudson River Project**

**Figure 2-9h Spring 2006  
fish sampling locations.**

GENbmp:112 March 2007





**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

**GRAPHIC SCALE**

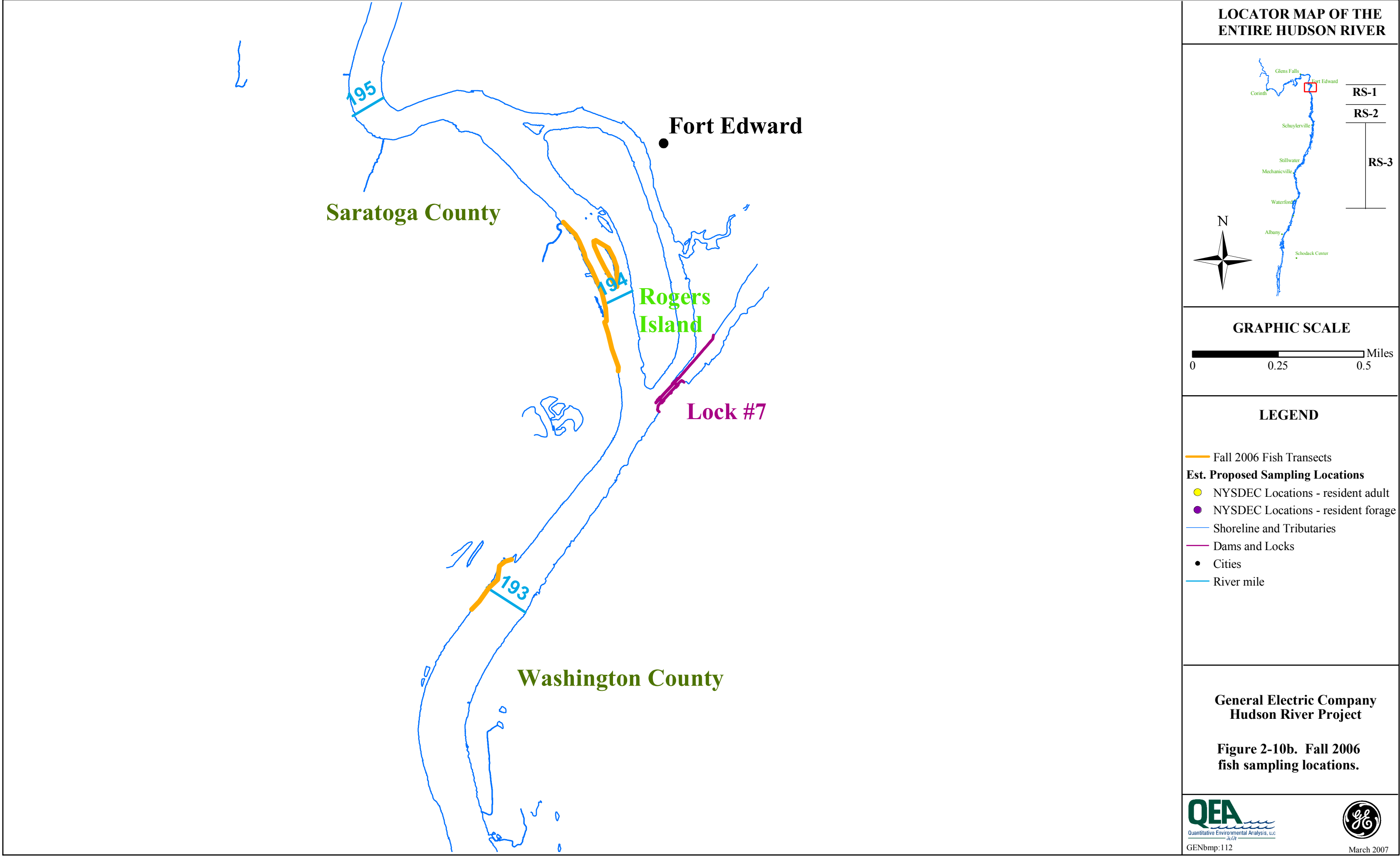
**LEGEND**

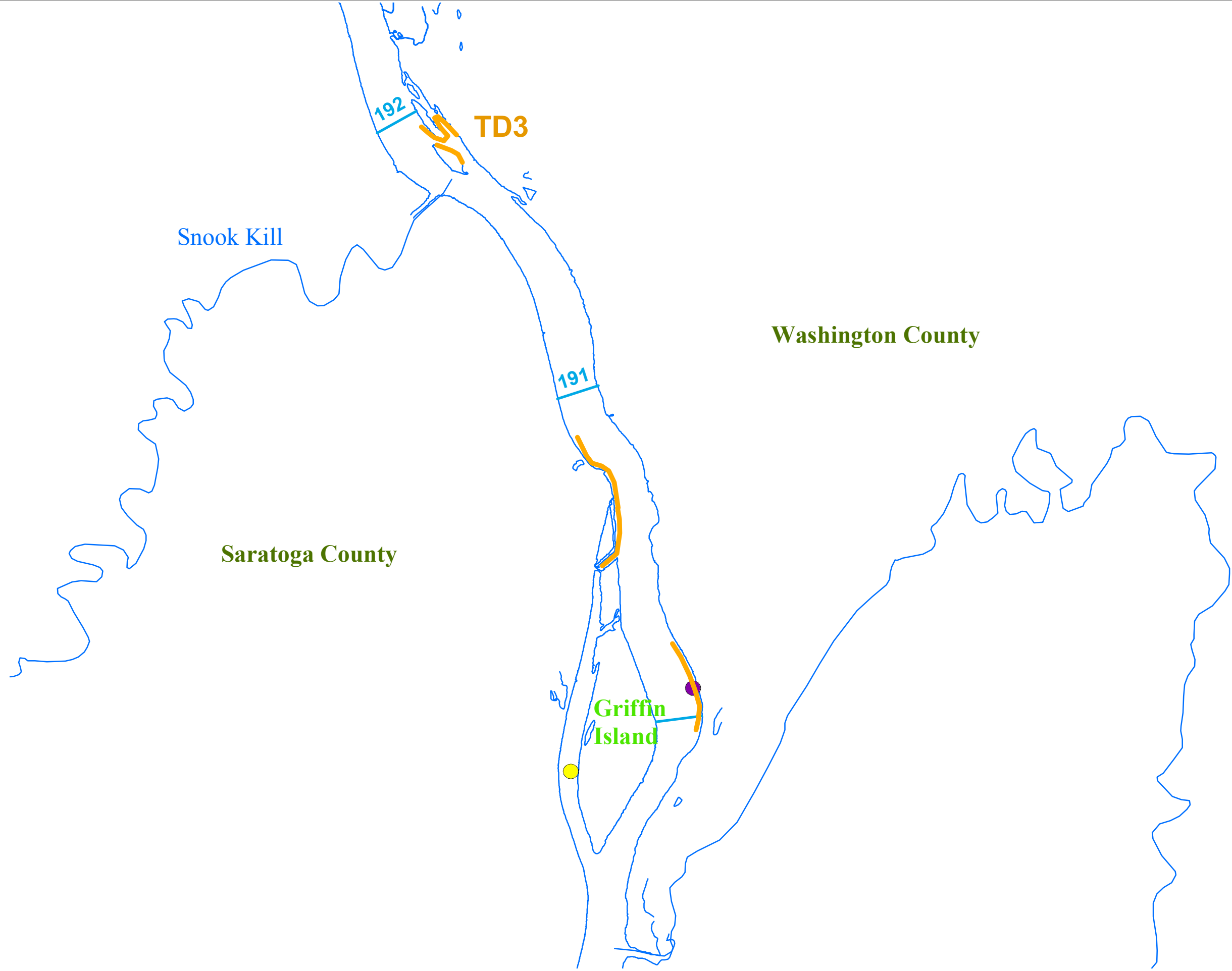
- Fall 2006 Fish Transects
- Est. Proposed Sampling Locations**
  - NYSDEC Locations - resident adult
  - NYSDEC Locations - resident forage
- Shoreline and Tributaries
- Dams and Locks
- Cities
- River mile

**General Electric Company  
Hudson River Project**  
  
**Figure 2-10a. Fall 2006  
fish sampling locations.**

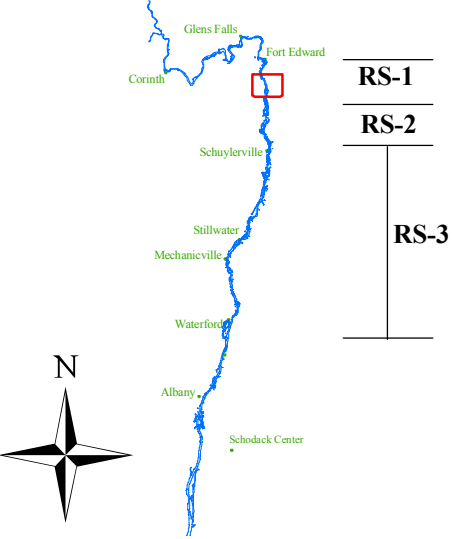
GENbmp:112

March 2007

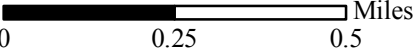




**LOCATOR MAP OF THE  
ENTIRE HUDSON RIVER**



**GRAPHIC SCALE**



**LEGEND**

- Fall 2006 Fish Transects
- Est. Proposed Sampling Locations**
  - NYSDEC Locations - resident adult
  - NYSDEC Locations - resident forage
- Shoreline and Tributaries
- Dams and Locks
- Cities
- River mile

**General Electric Company  
Hudson River Project**

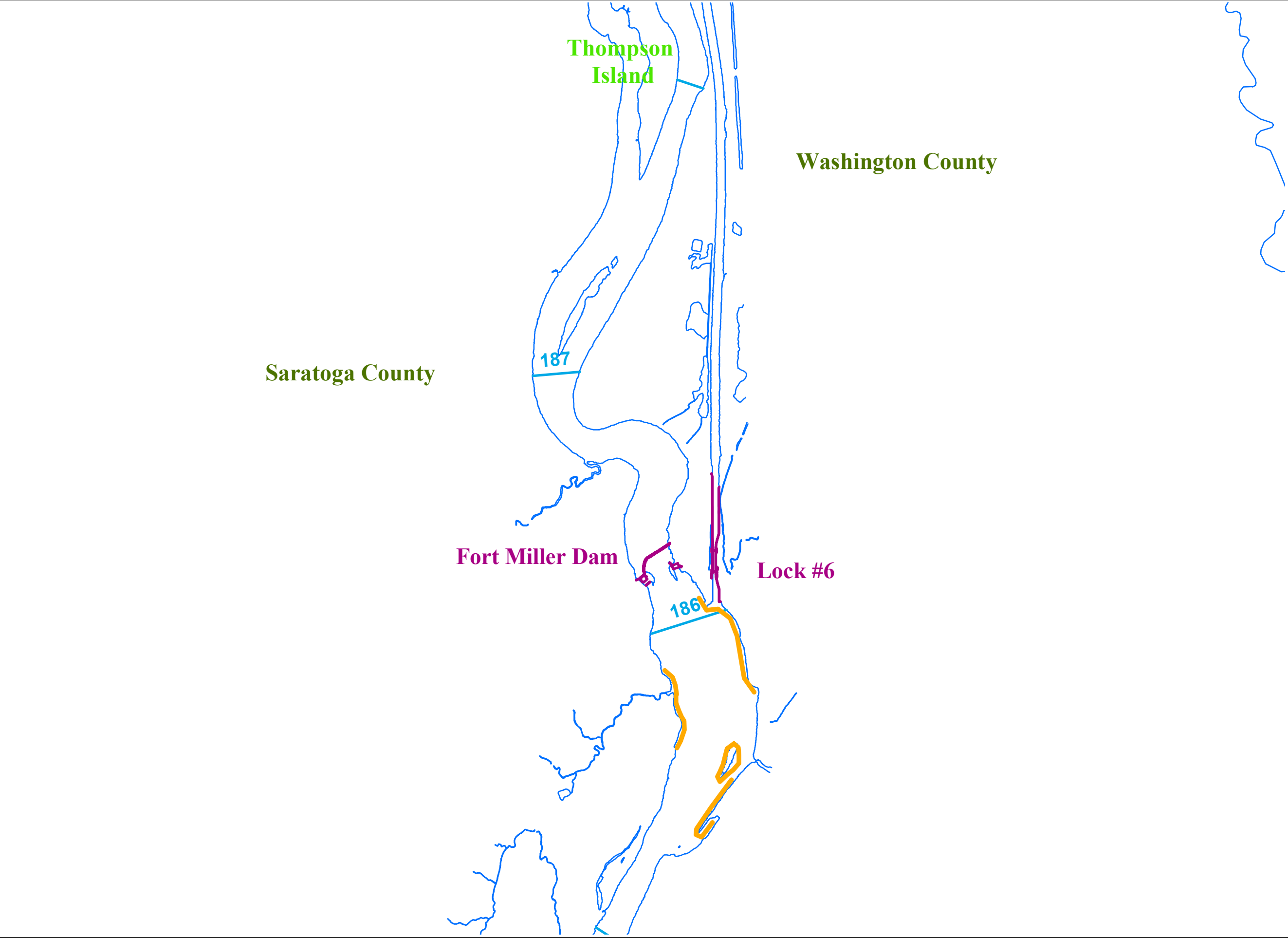
**Figure 2-10c. Fall 2006  
fish sampling locations.**



GENbmp:112



March 2007



**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

RS-1

RS-2

RS-3

00.250.5 Miles

**LEGEND**

Fall 2006 Fish Transects

**Est. Proposed Sampling Locations**

NYSDEC Locations - resident adult

NYSDEC Locations - resident forage

Shoreline and Tributaries

Dams and Locks

Cities

River mile

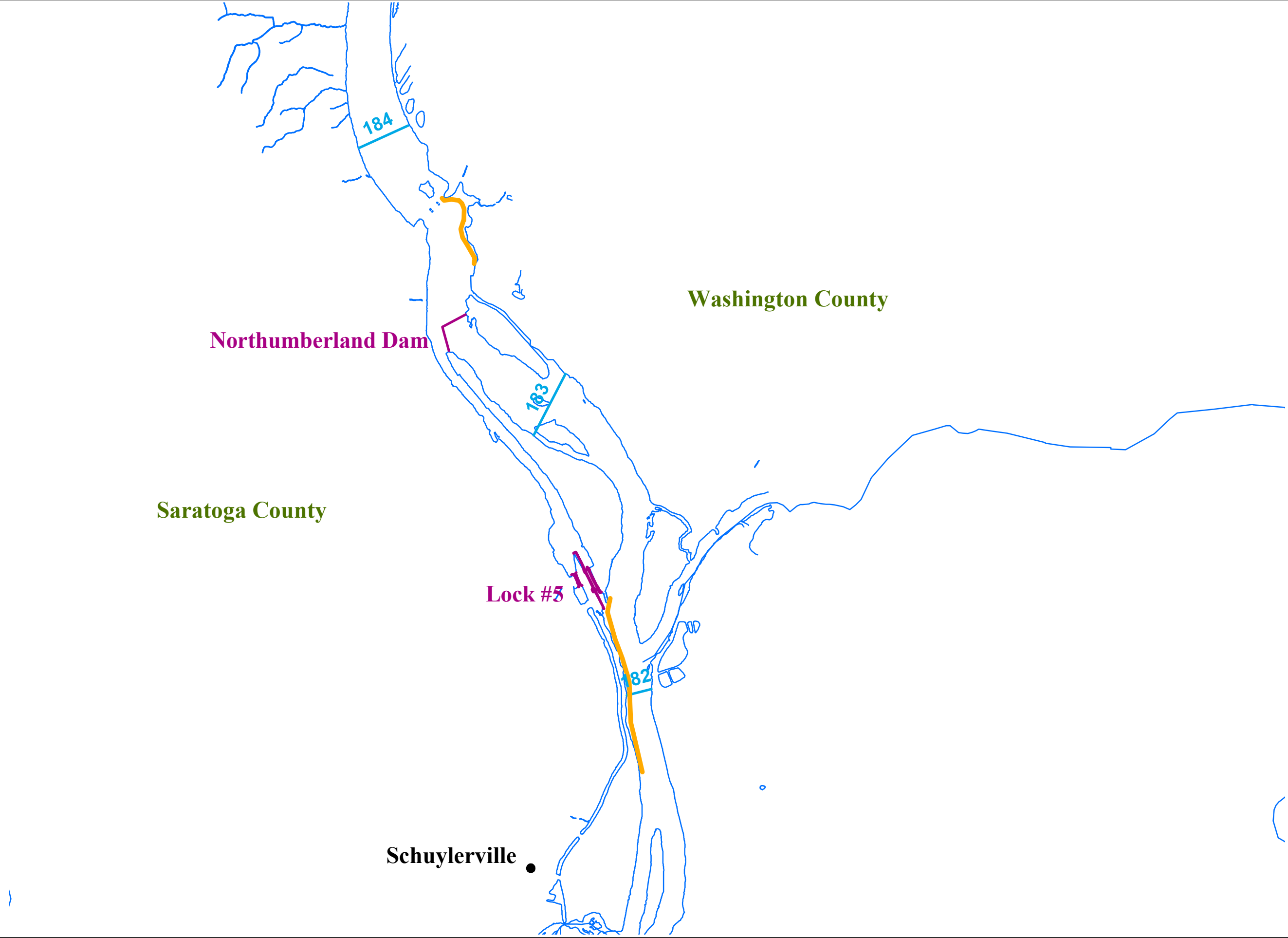
**General Electric Company  
Hudson River Project**

**Figure 2-10d. Fall 2006  
fish sampling locations.**

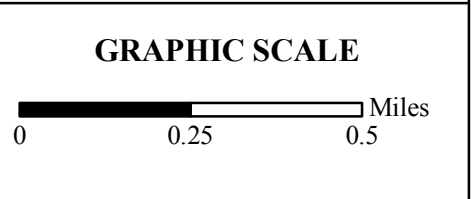
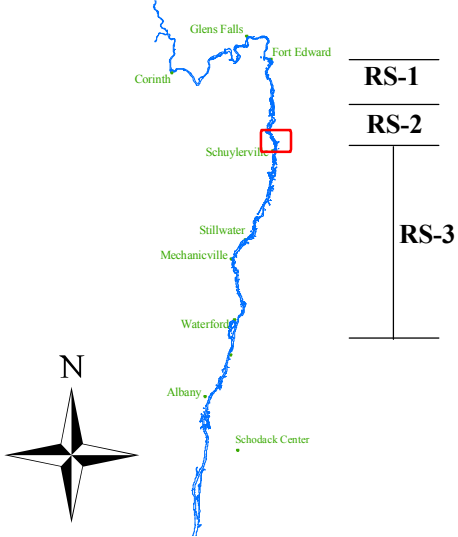
Quantitative Environmental Analysis, LLC

March 2007

GENbmp:112



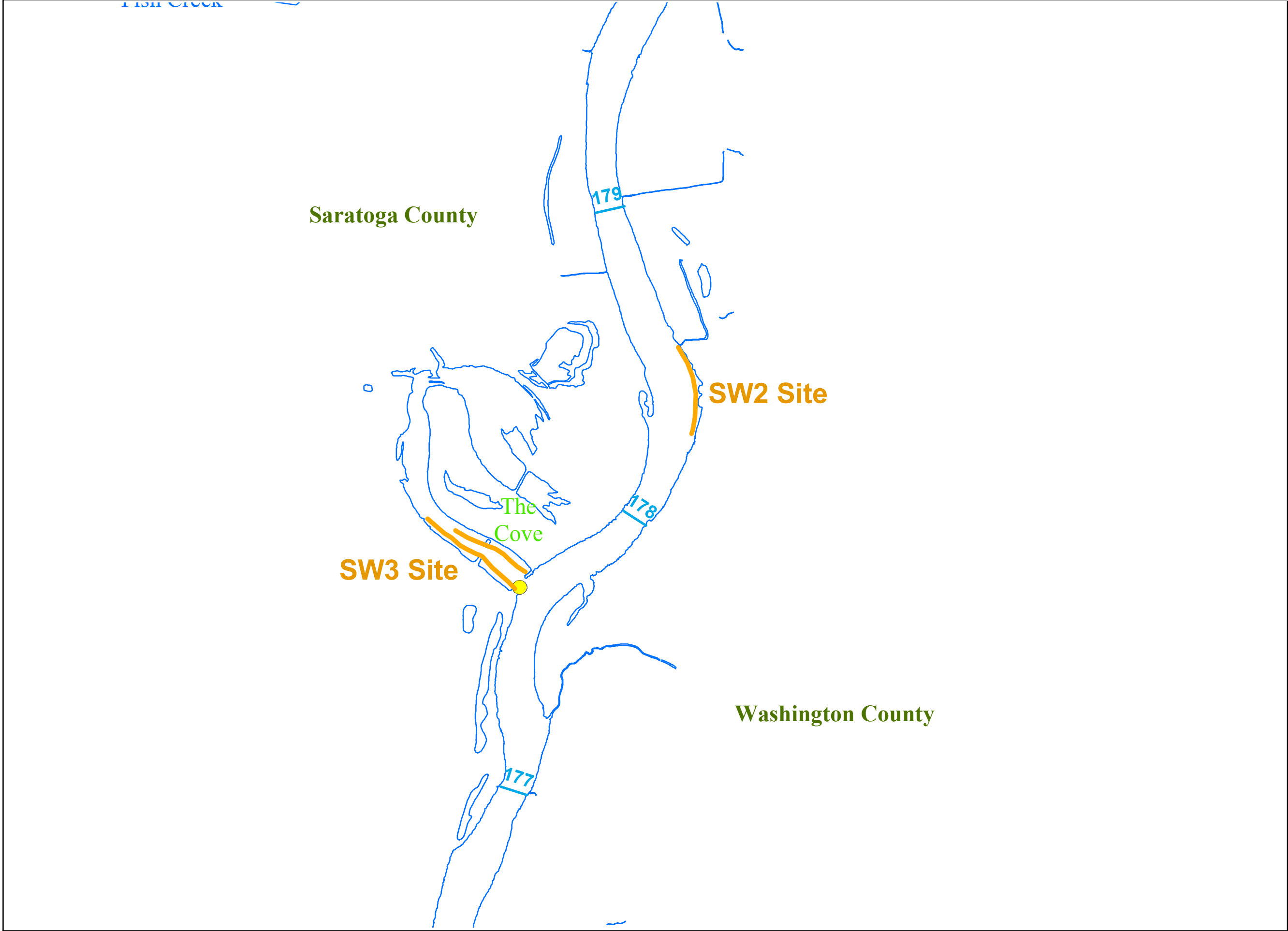
LOCATOR MAP OF THE ENTIRE HUDSON RIVER



- LEGEND
- Fall 2006 Fish Transects
  - Est. Proposed Sampling Locations
    - NYSDEC Locations - resident adult
    - NYSDEC Locations - resident forage
  - Shoreline and Tributaries
  - Dams and Locks
  - Cities
  - River mile

General Electric Company  
Hudson River Project

Figure 2-10e. Fall 2006  
fish sampling locations.



**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

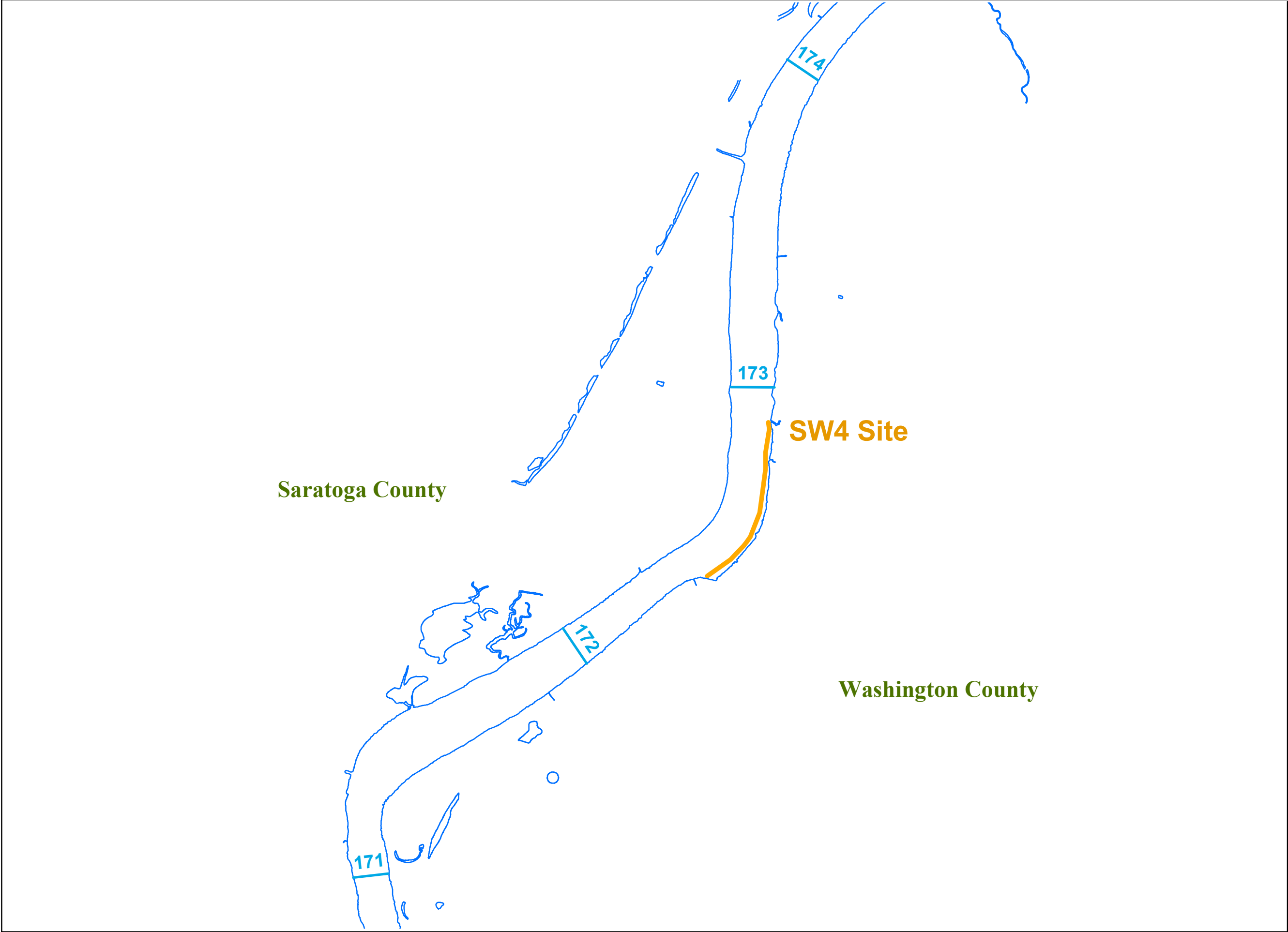
**GRAPHIC SCALE**

**LEGEND**

- Fall 2006 Fish Transects
- Est. Proposed Sampling Locations**
  - NYSDEC Locations - resident adult
  - NYSDEC Locations - resident forage
- Shoreline and Tributaries
- Dams and Locks
- Cities
- River mile

**General Electric Company  
Hudson River Project**  
  
**Figure 2-10f. Fall 2006  
fish sampling locations.**

GENbmp:112March 2007



**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

**GRAPHIC SCALE**

0 0.25 0.5 Miles

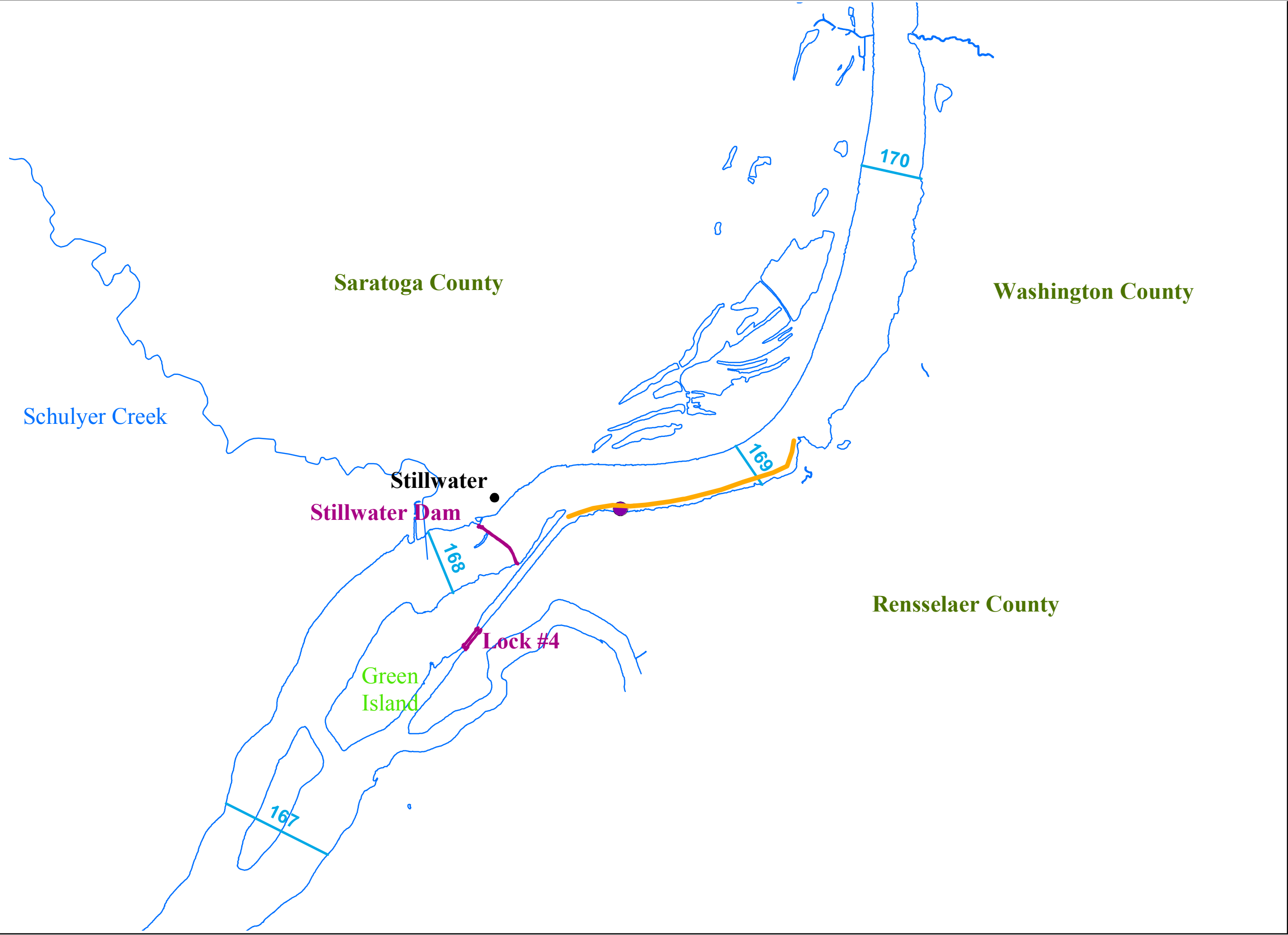
**LEGEND**

- Fall 2006 Fish Transects
- Est. Proposed Sampling Locations**
- NYSDEC Locations - resident adult
- NYSDEC Locations - resident forage
- Shoreline and Tributaries
- Dams and Locks
- Cities
- River mile

**General Electric Company  
Hudson River Project**

**Figure 2-10g. Fall 2006  
fish sampling locations.**

GENbmp:112 March 2007



**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

N

00.250.5 Miles

**GRAPHIC SCALE**

00.250.5 Miles

**LEGEND**

Fall 2006 Fish Transects

**Est. Proposed Sampling Locations**

NYSDEC Locations - resident adult

NYSDEC Locations - resident forage

Shoreline and Tributaries

Dams and Locks

Cities

River mile

**General Electric Company  
Hudson River Project**

**Figure 2-10h. Fall 2006  
fish sampling locations.**

**QEA**

Quantitative Environmental Analysis, LLC

GENbmp:112

March 2007

DR/LC - \\Frodo\D\_Drive\QEA\_Syracuse\Jobs\GENrem\GIS\maps\ArcGIS8\_maps\Baseline\_Monitoring\BMP\_Fall2006\_Fish\_Locations.mxd



**LOCATOR MAP OF THE ENTIRE HUDSON RIVER**

**GRAPHIC SCALE**

0 0.25 0.5 Miles

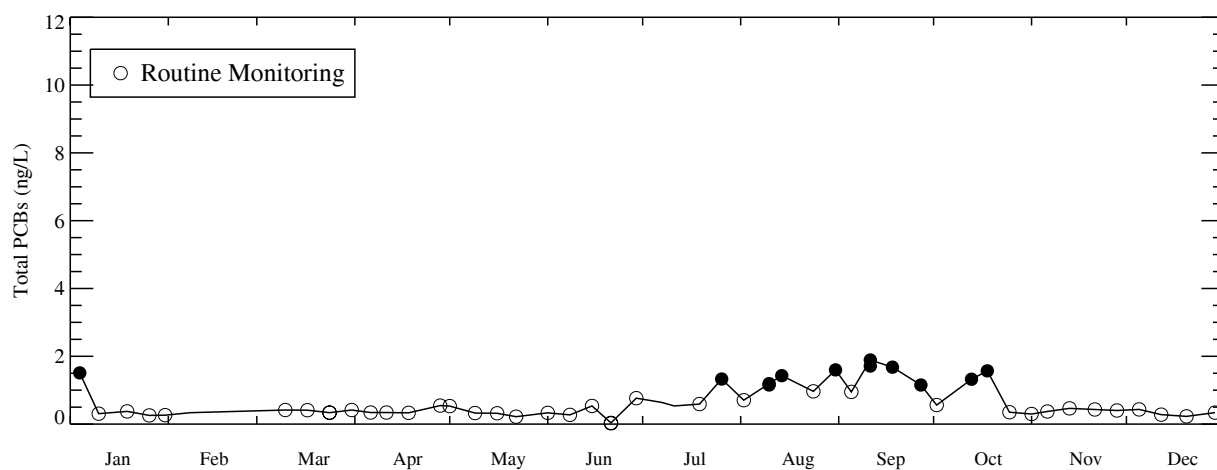
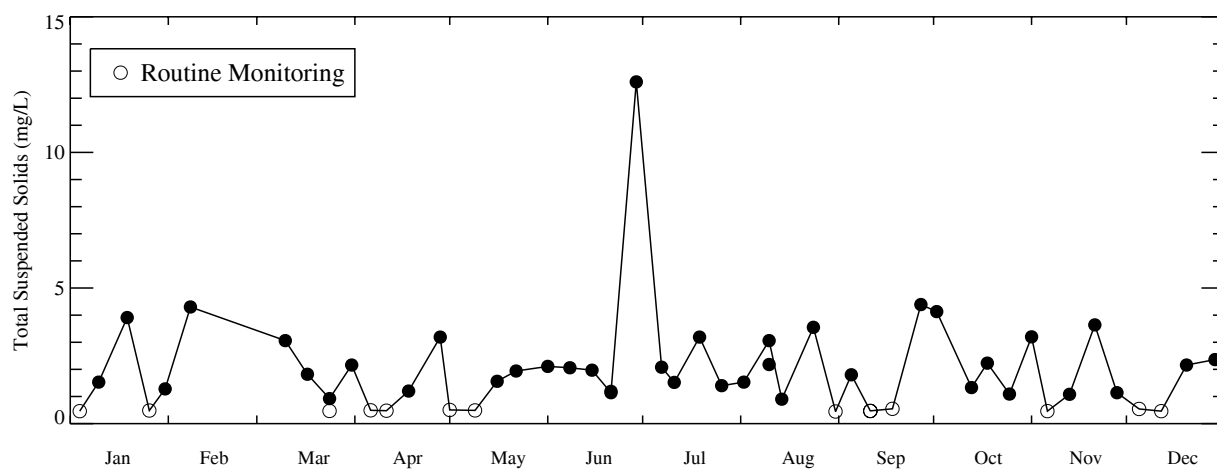
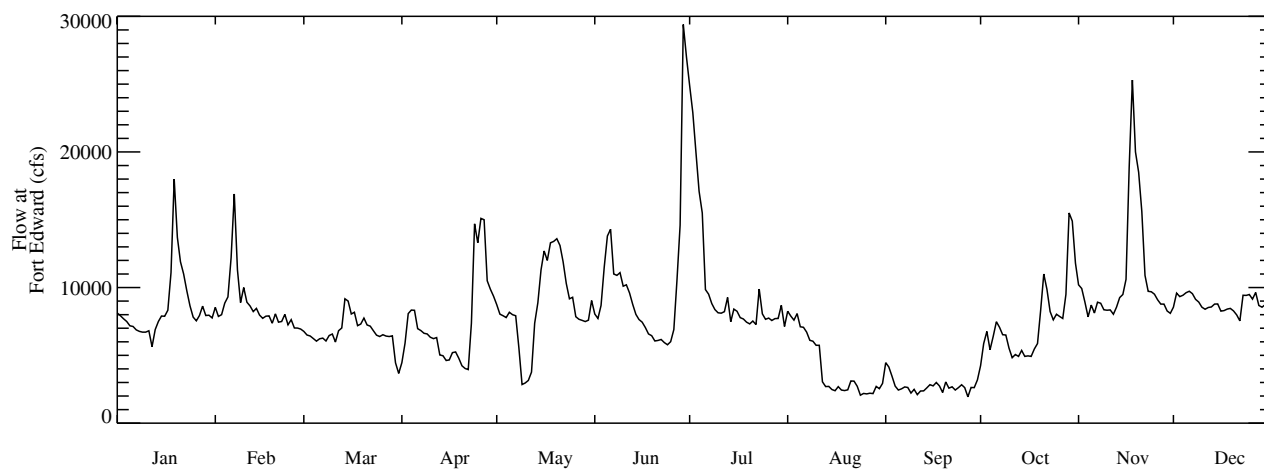
**LEGEND**

- Fall 2006 Fish Transects
- Est. Proposed Sampling Locations**
- NYSDEC Locations - resident adult
- NYSDEC Locations - resident forage
- Shoreline and Tributaries
- Dams and Locks
- Cities
- River mile

**General Electric Company  
Hudson River Project**

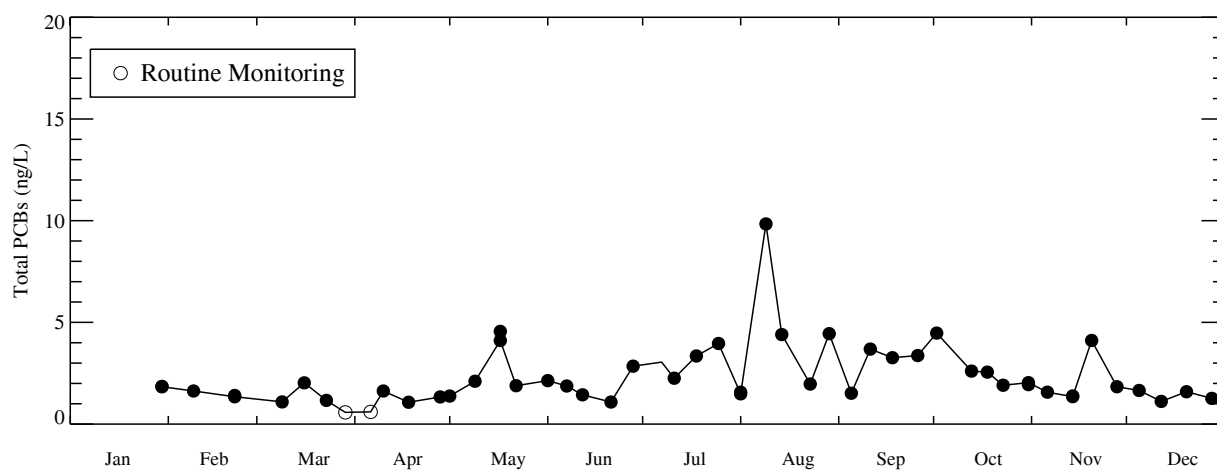
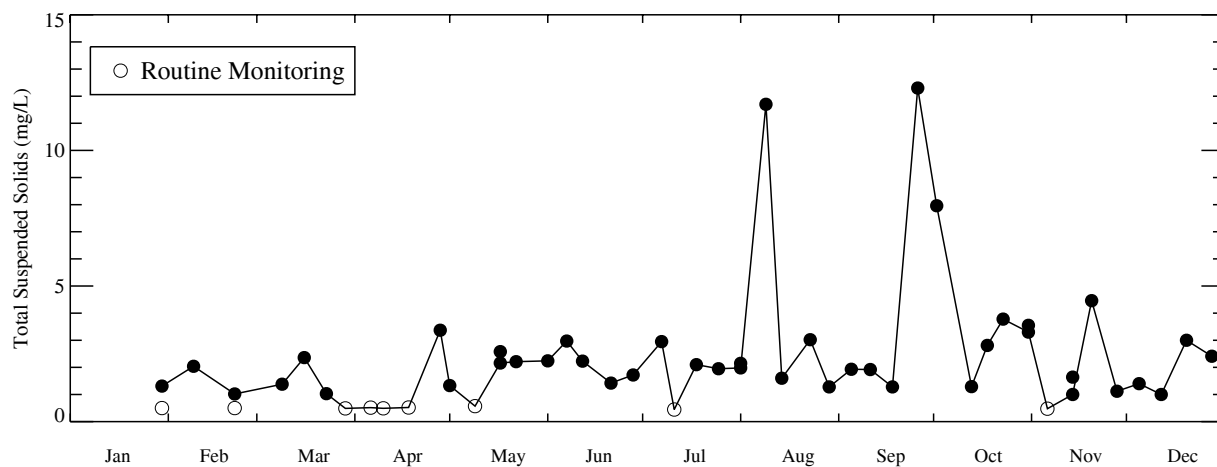
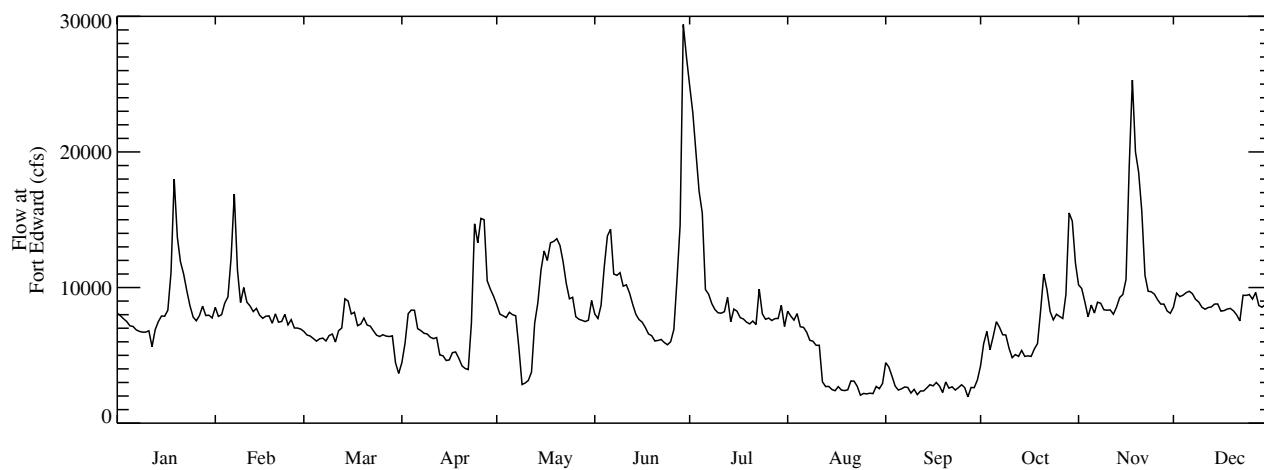
**Figure 2-10i. Fall 2006  
fish sampling locations.**

GENbmp:112 March 2007



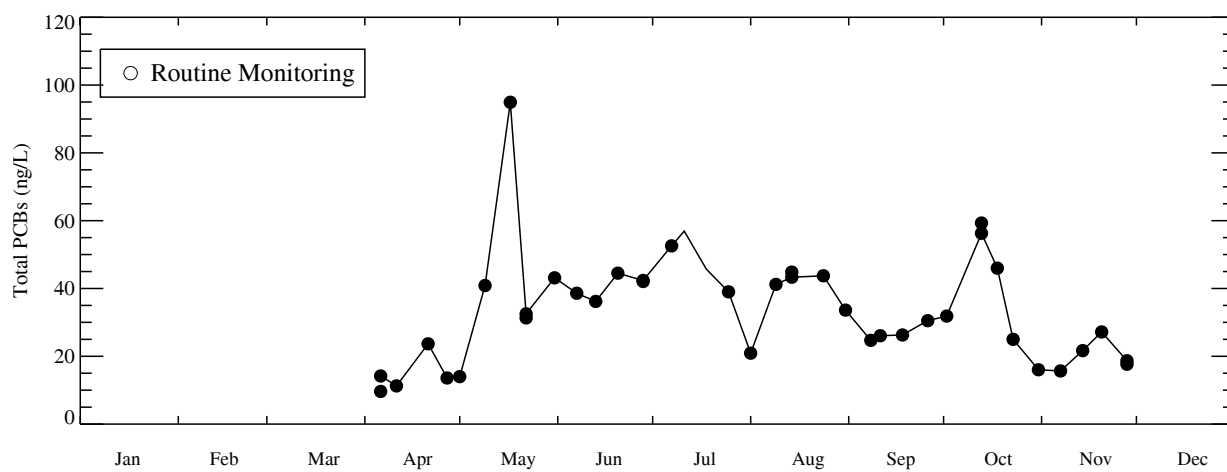
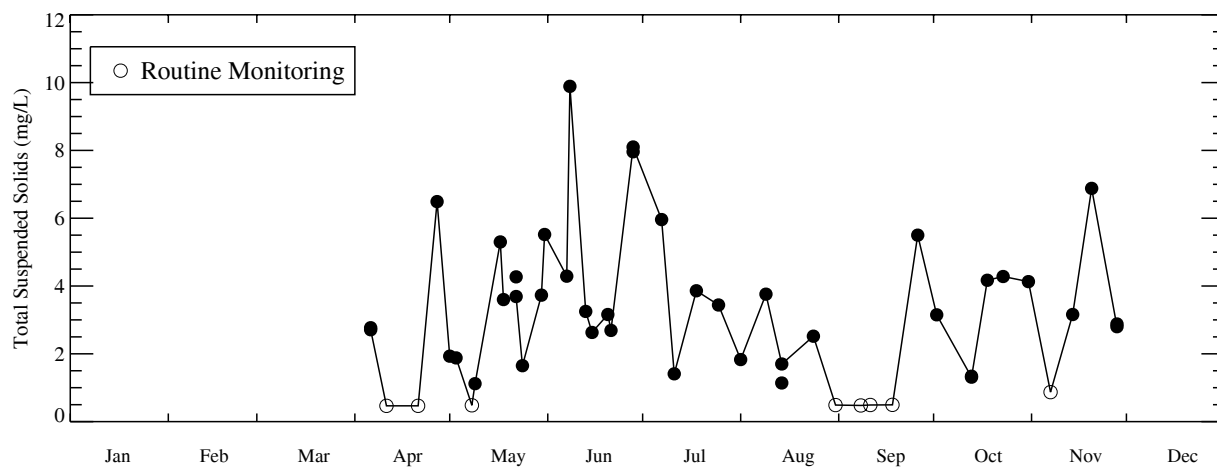
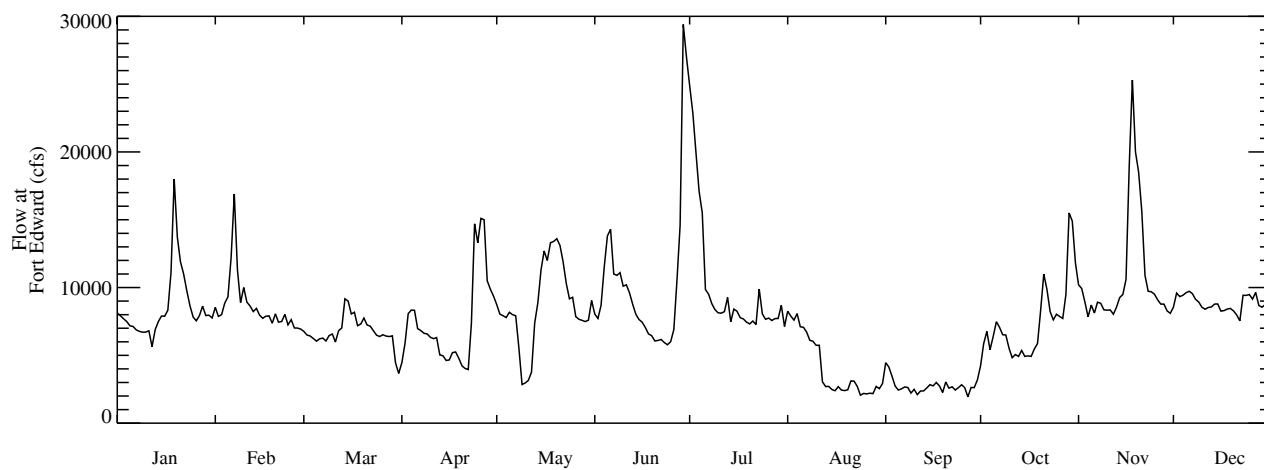
**Figure 4-1. 2006 Temporal profiles of PCB and TSS results at Bakers Falls.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



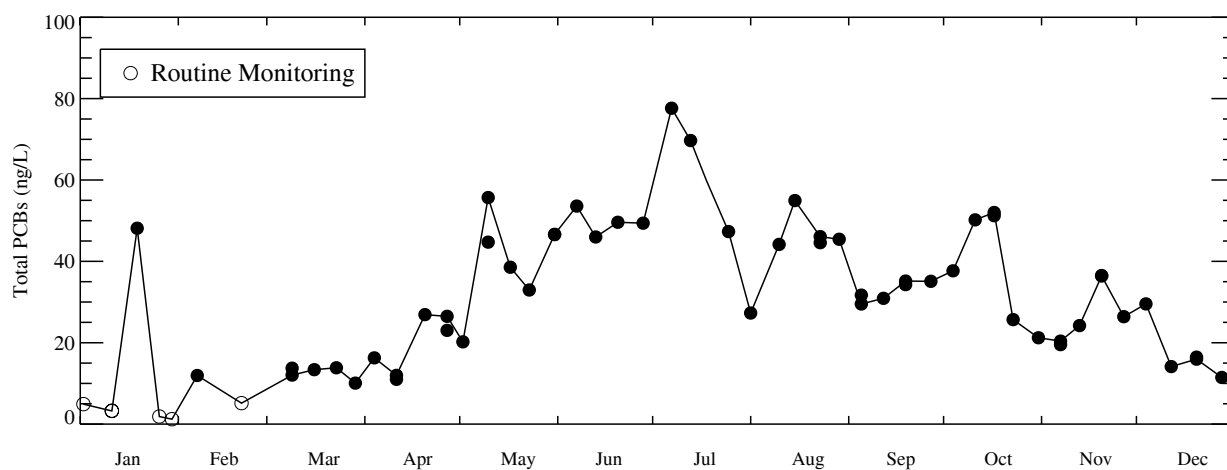
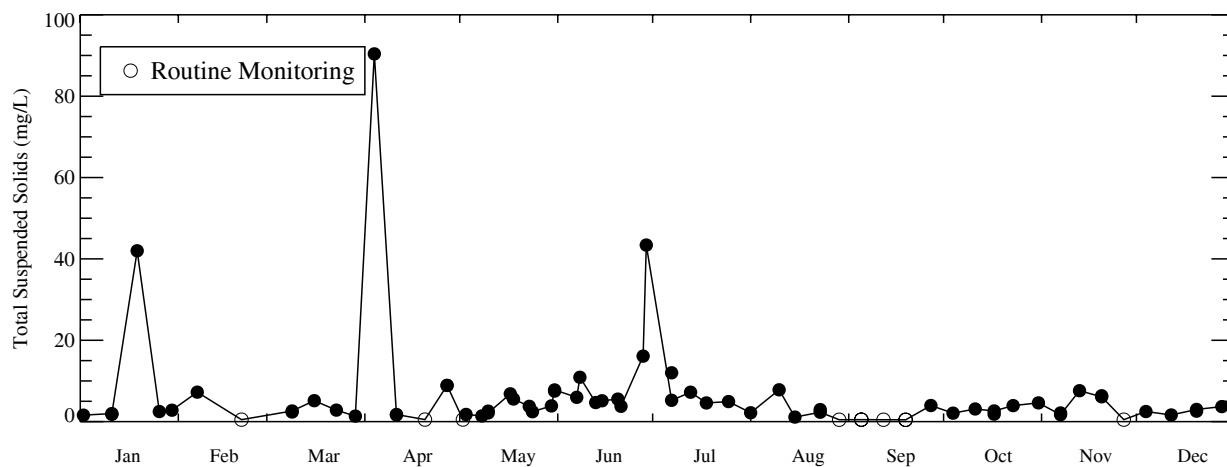
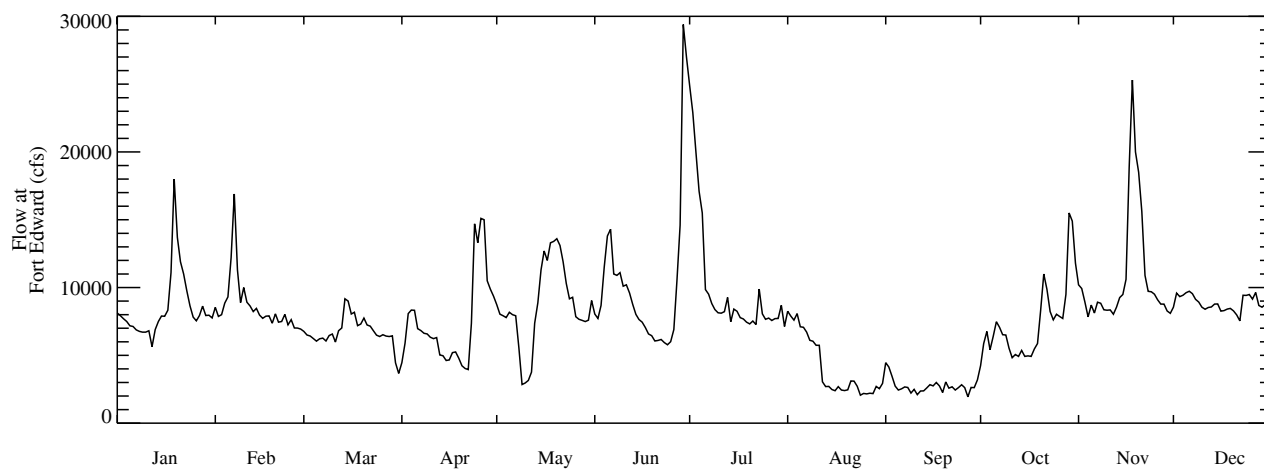
**Figure 4-2. 2006 Temporal profiles of PCB and TSS results at Rogers Island.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



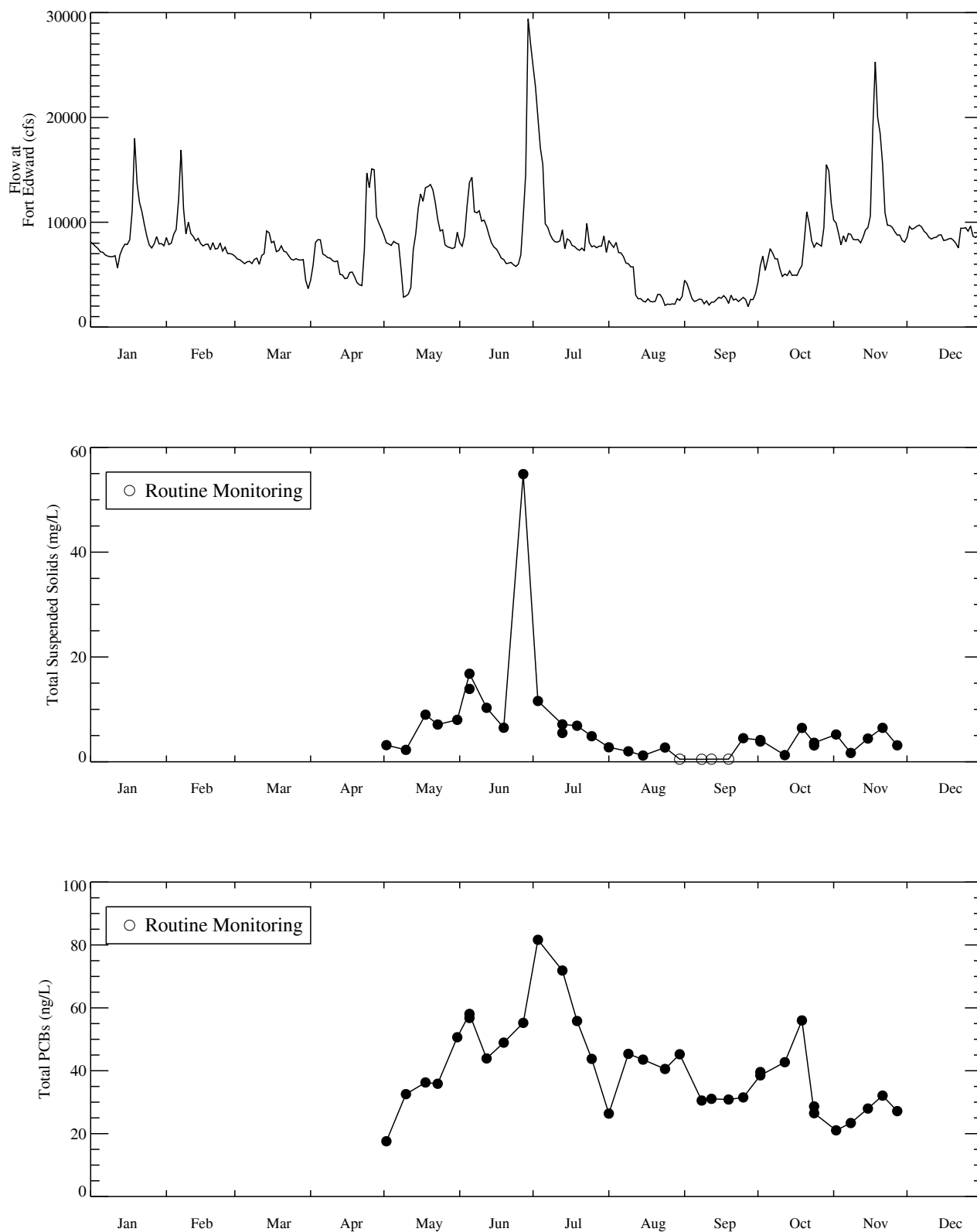
**Figure 4-3. 2006 Temporal profiles of PCB and TSS results at Thompson Island.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



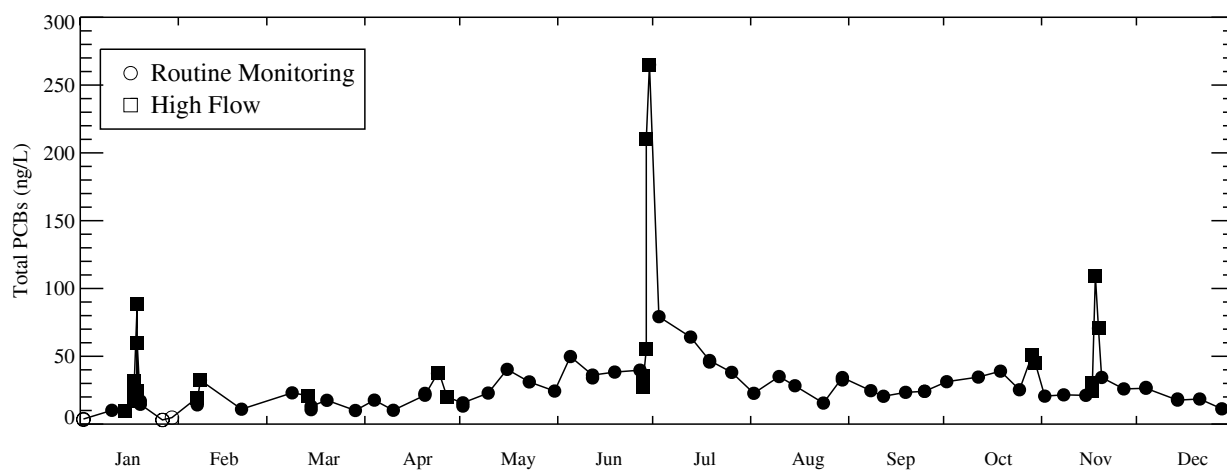
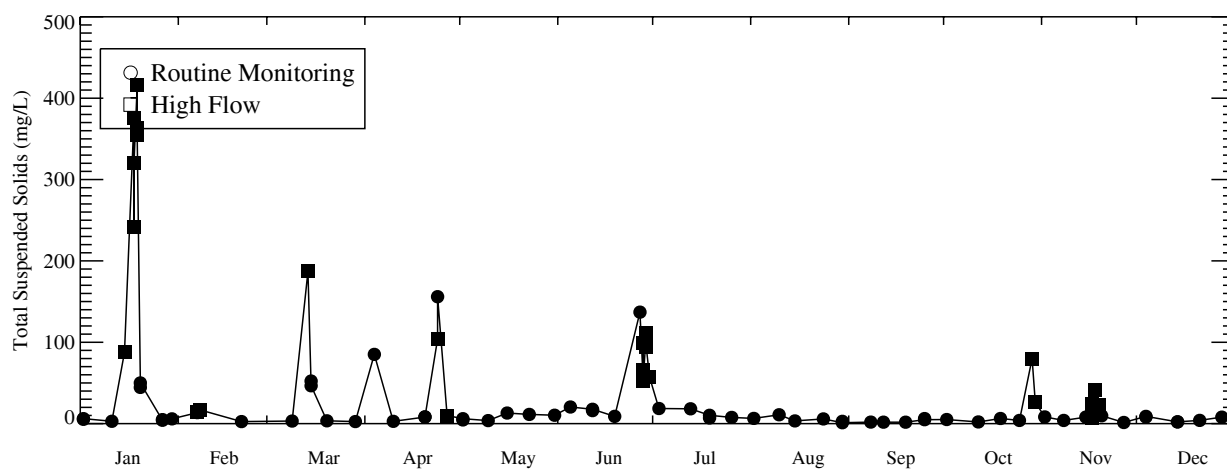
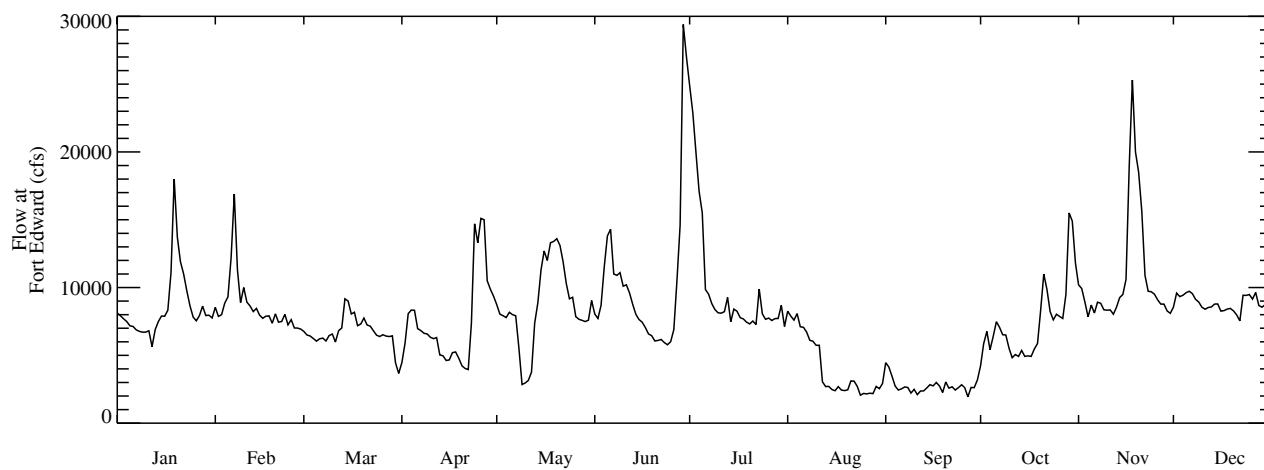
**Figure 4-4. 2006 Temporal profiles of PCB and TSS results at Schuylerville.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



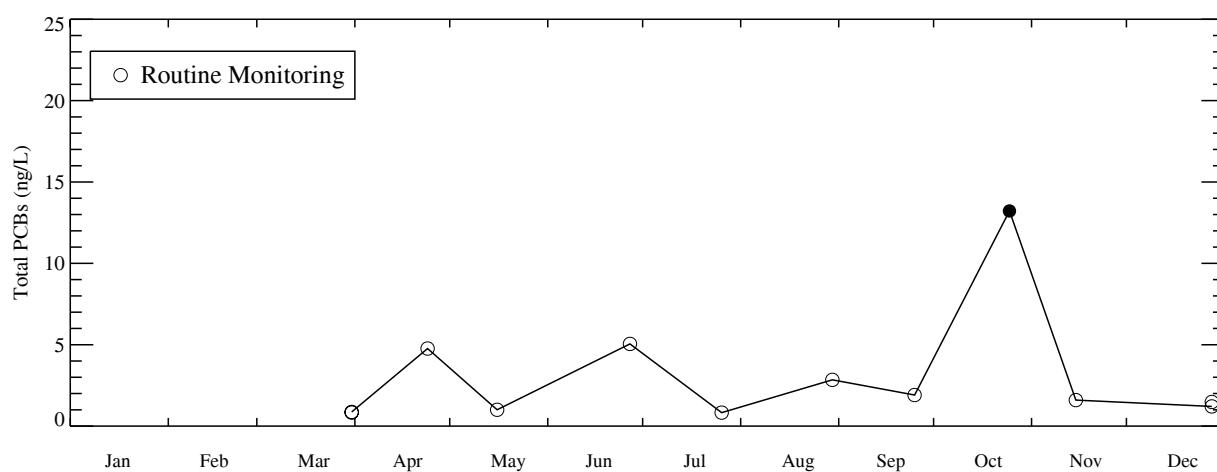
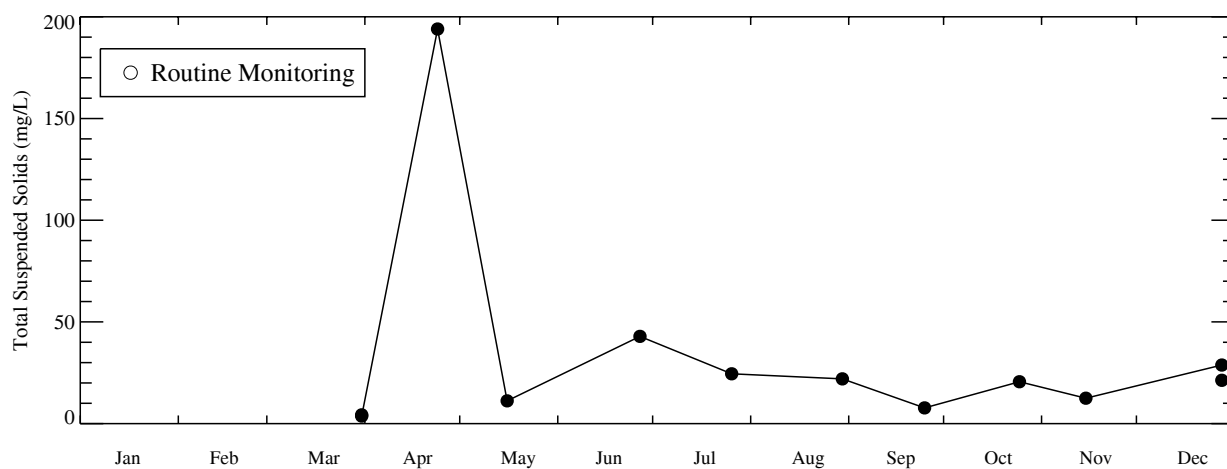
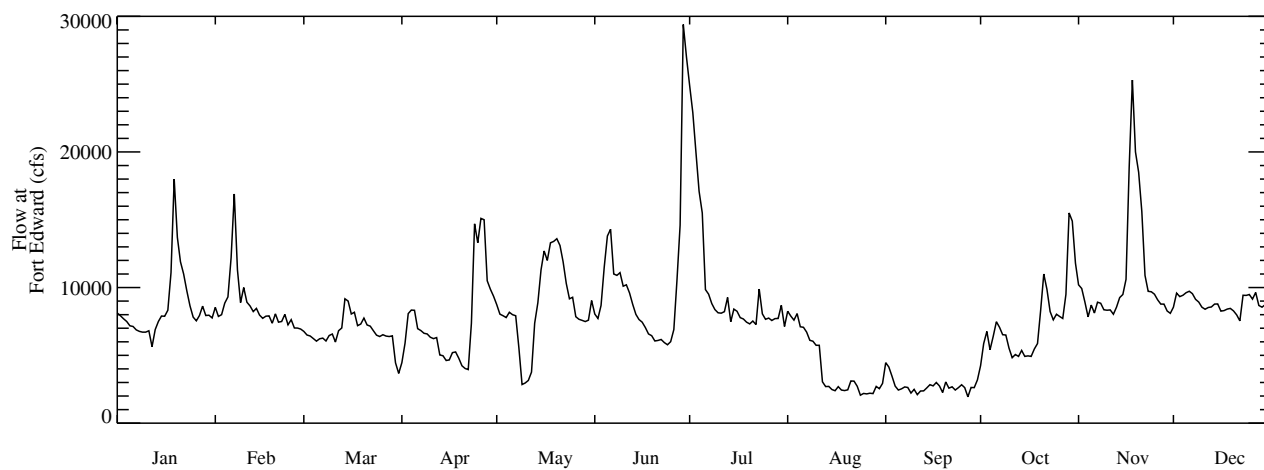
**Figure 4-5. 2006 Temporal profiles of PCB and TSS results at Stillwater.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



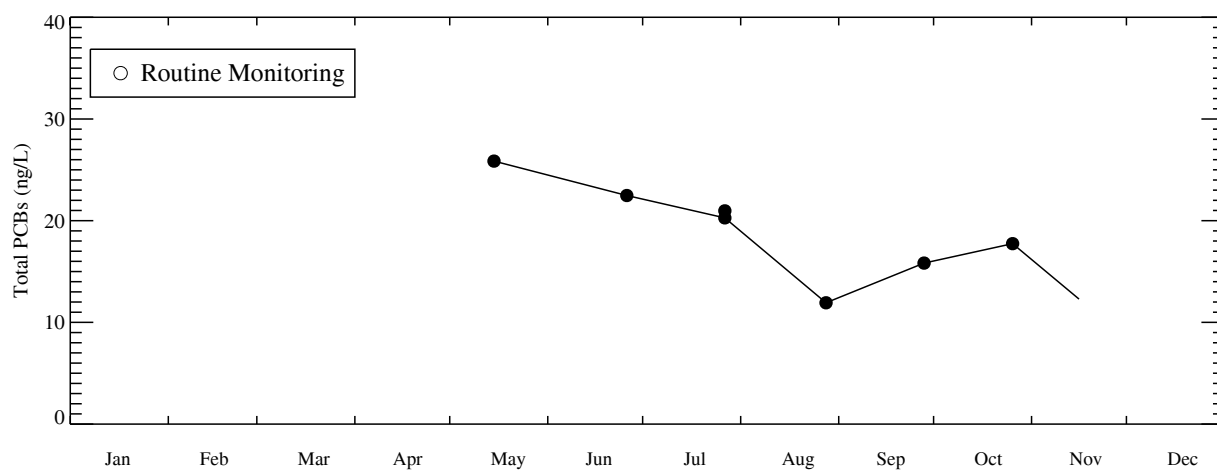
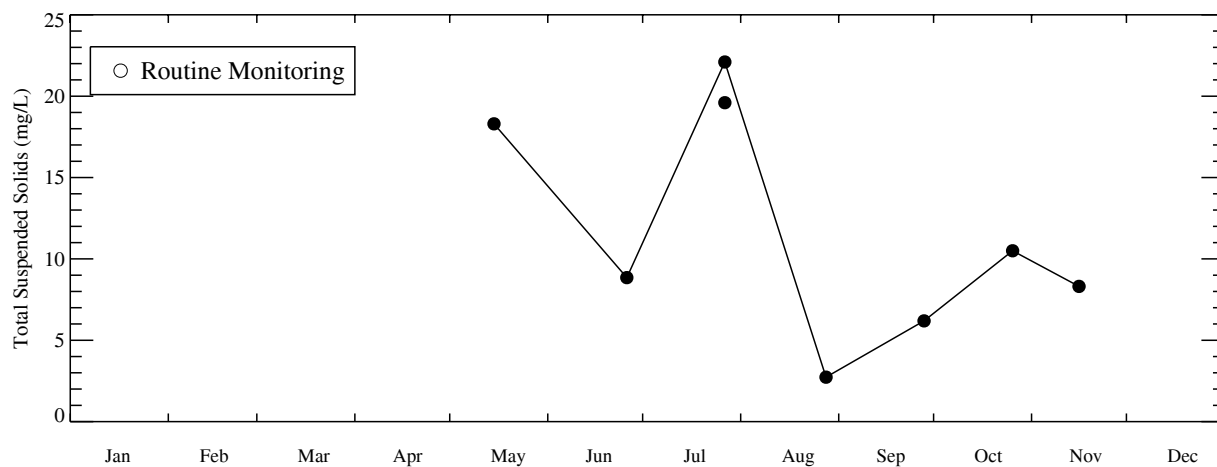
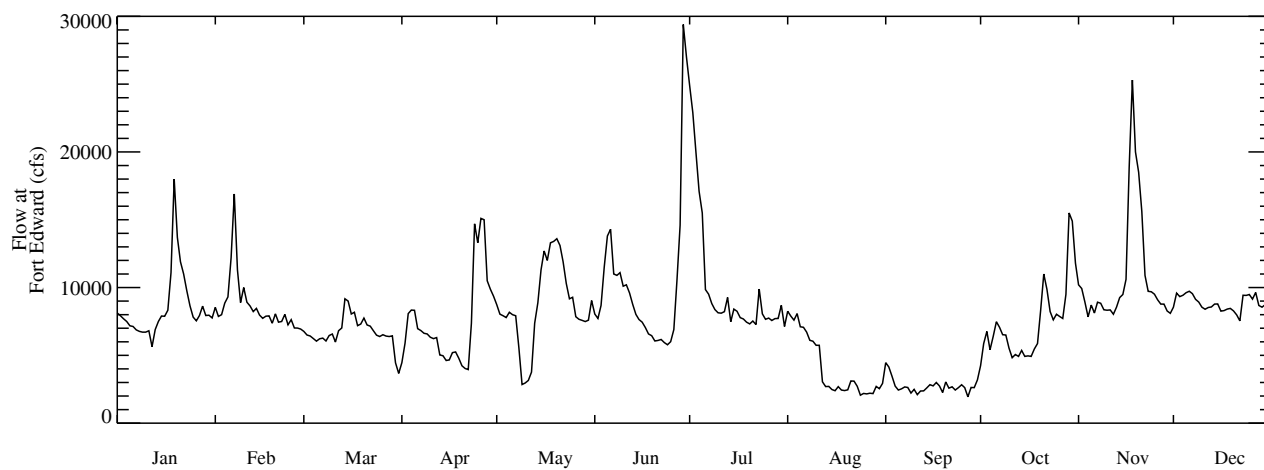
**Figure 4-6. 2006 Temporal profiles of PCB and TSS results at Waterford.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



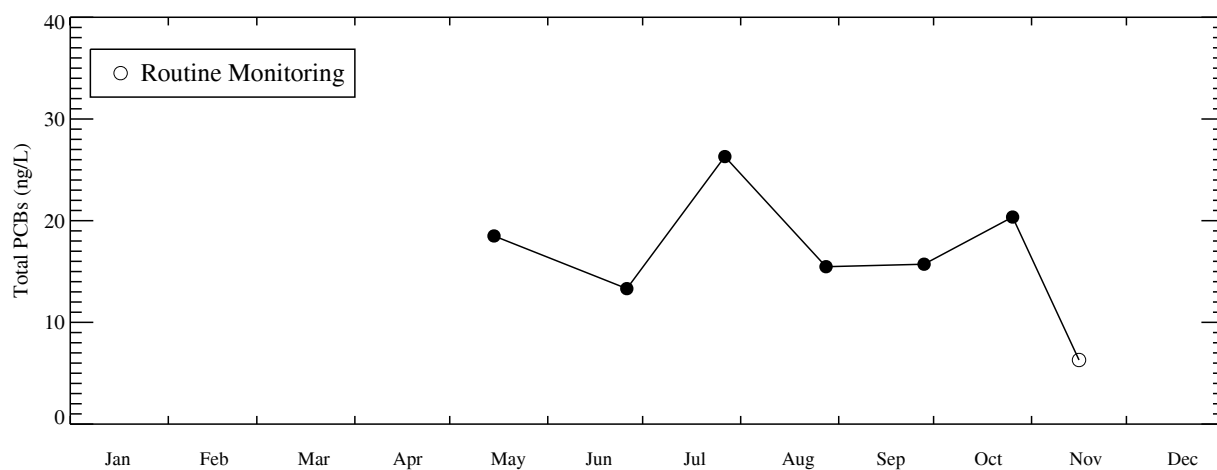
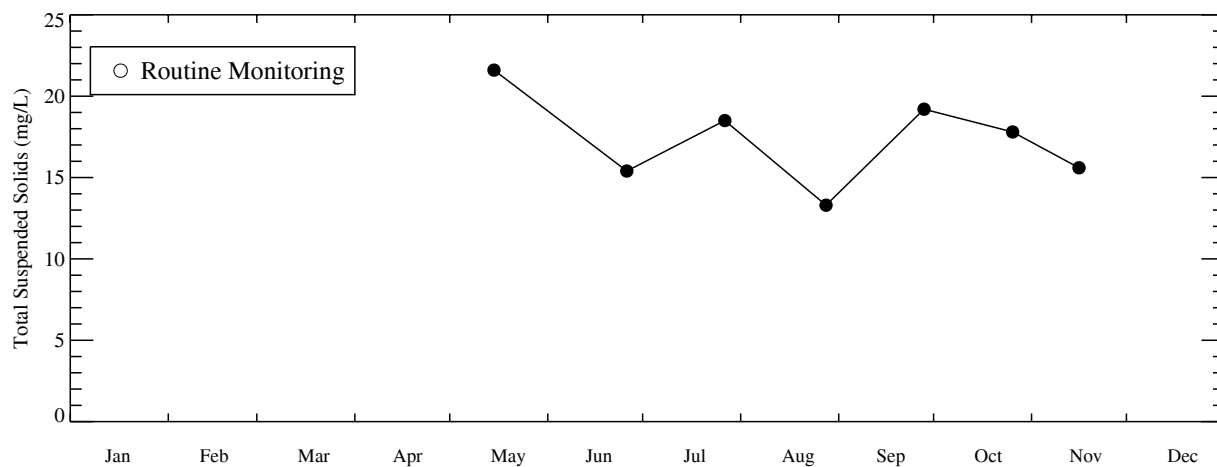
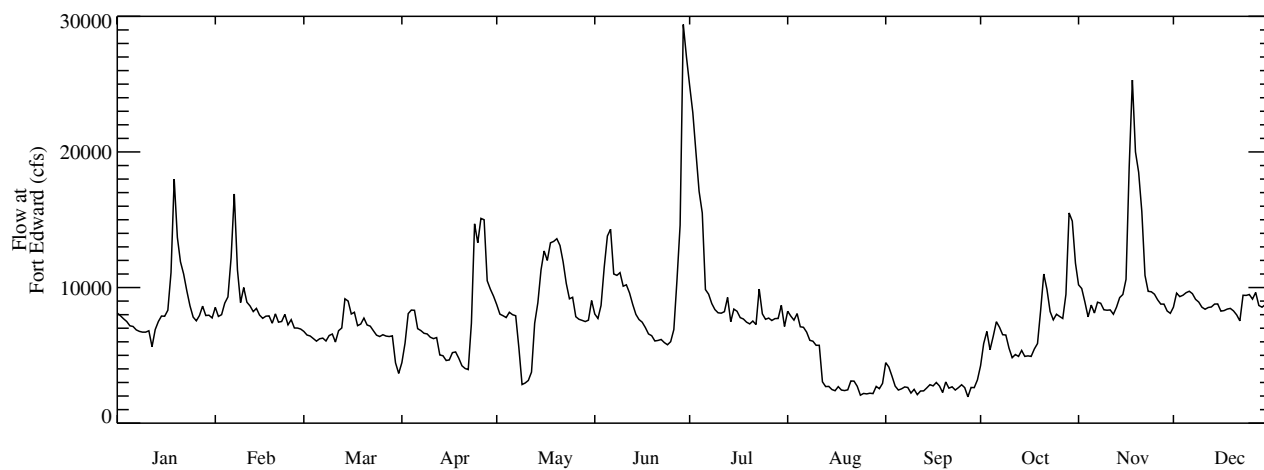
**Figure 4-7. 2006 Temporal profiles of PCB and TSS results at Mohawk River.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



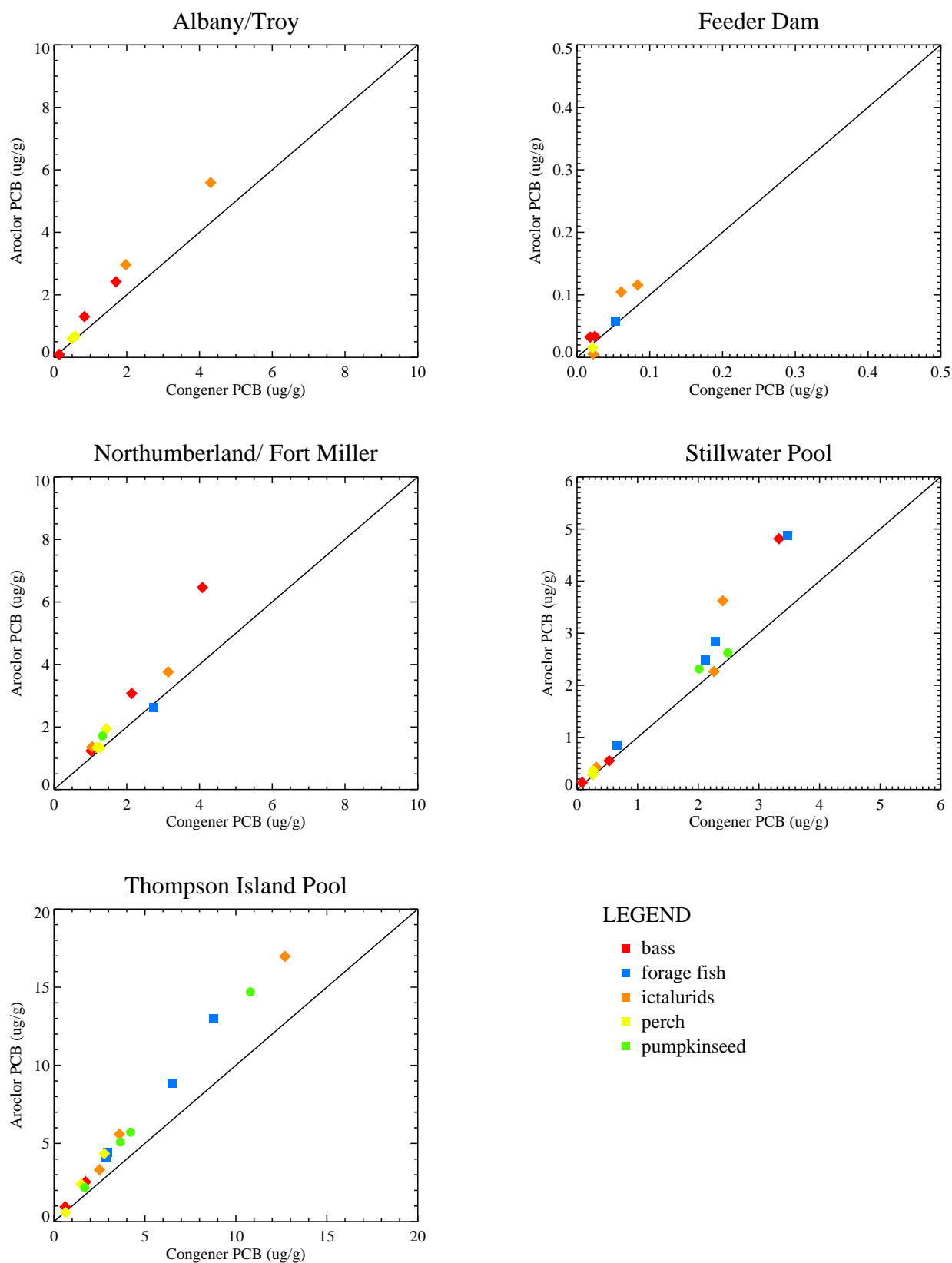
**Figure 4-8. 2006 Temporal profiles of PCB and TSS results at Albany.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



**Figure 4-9. 2006 Temporal profiles of PCB and TSS results at Poughkeepsie.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



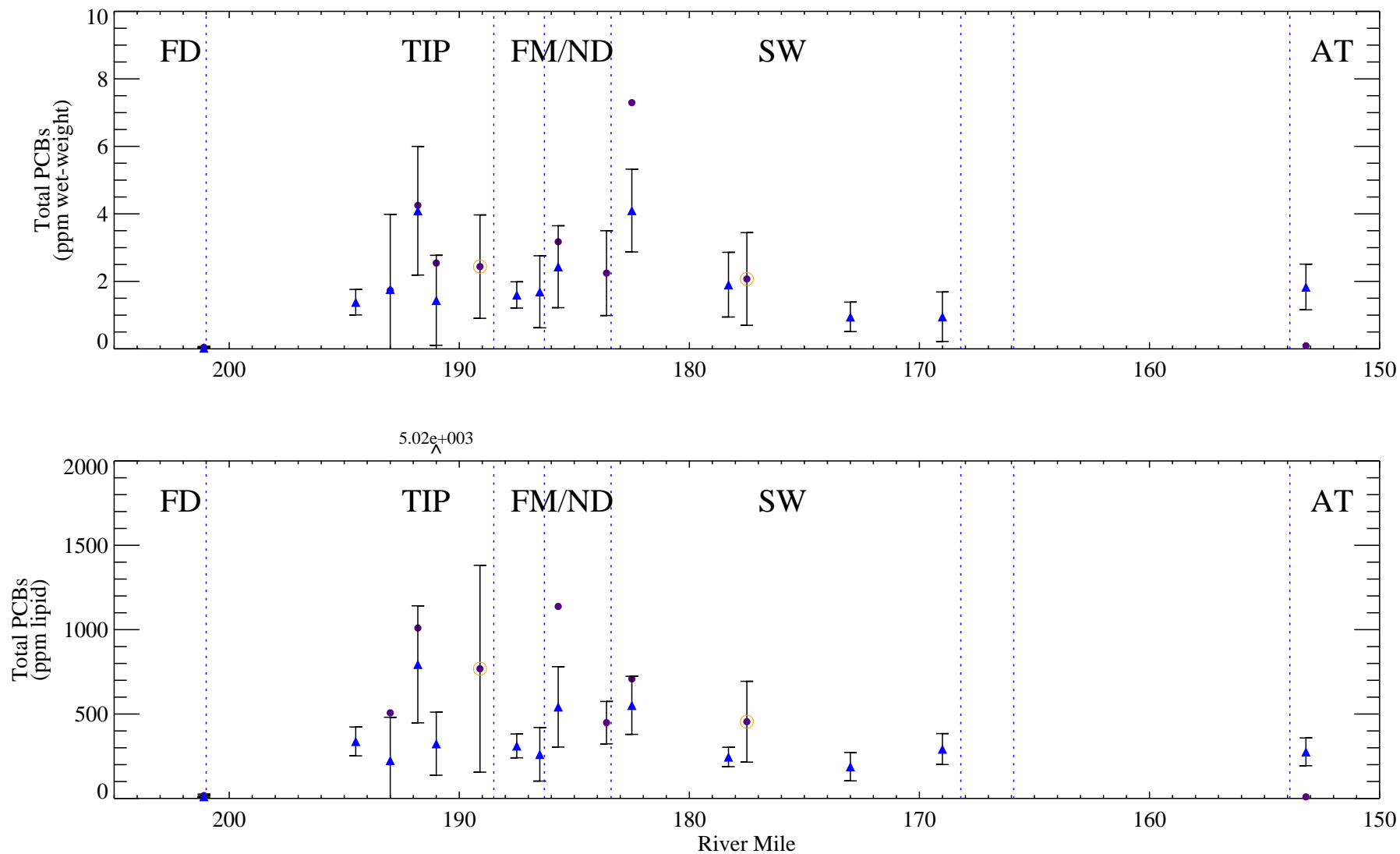
**Figure 5-1. Comparison of congener-specific PCB data with Aroclor PCB in fish.**

*Non-detect values were set to half the method detection limit.*

*Prep: diamonds = SF; circles = whole body (individual); squares = whole body (composite).*

*Year: 2006.*

*Source: BMP (QEAExport\_Fish 03/08/2007).*



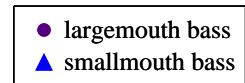
**Figure 5-2. Spatial patterns in PCB concentrations in black bass.**

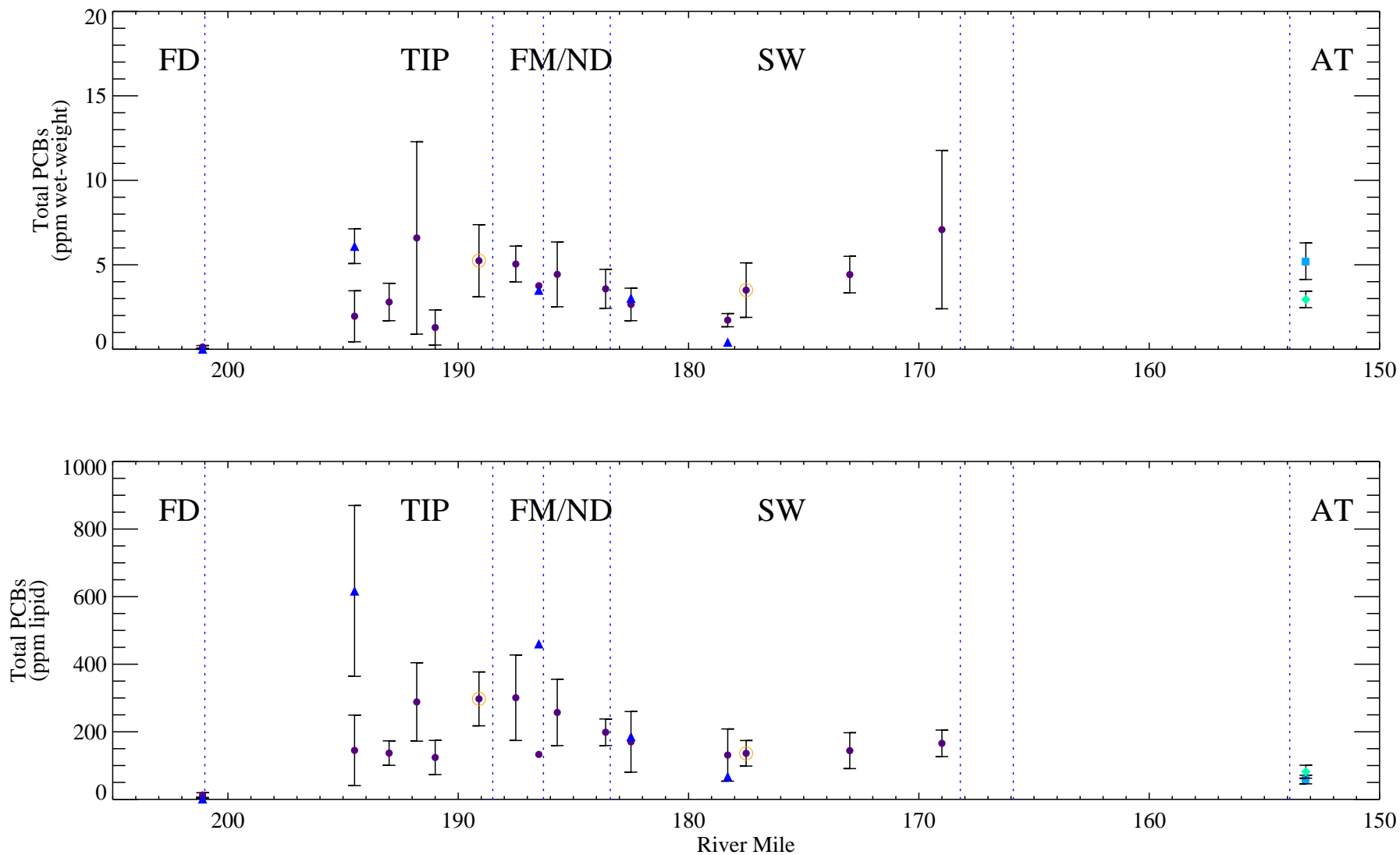
*Prep: fillet*

*Year: 2006.*

*Orange circles indicate historic sampling locations. Blue dotted lines indicate approximate dam locations.*

*Source: 2006 BMP (QEAExport\_Fish 03/08/2007).*





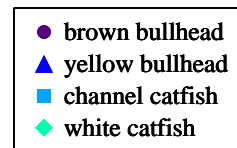
**Figure 5-3. Spatial patterns in PCB concentrations in ictalurids.**

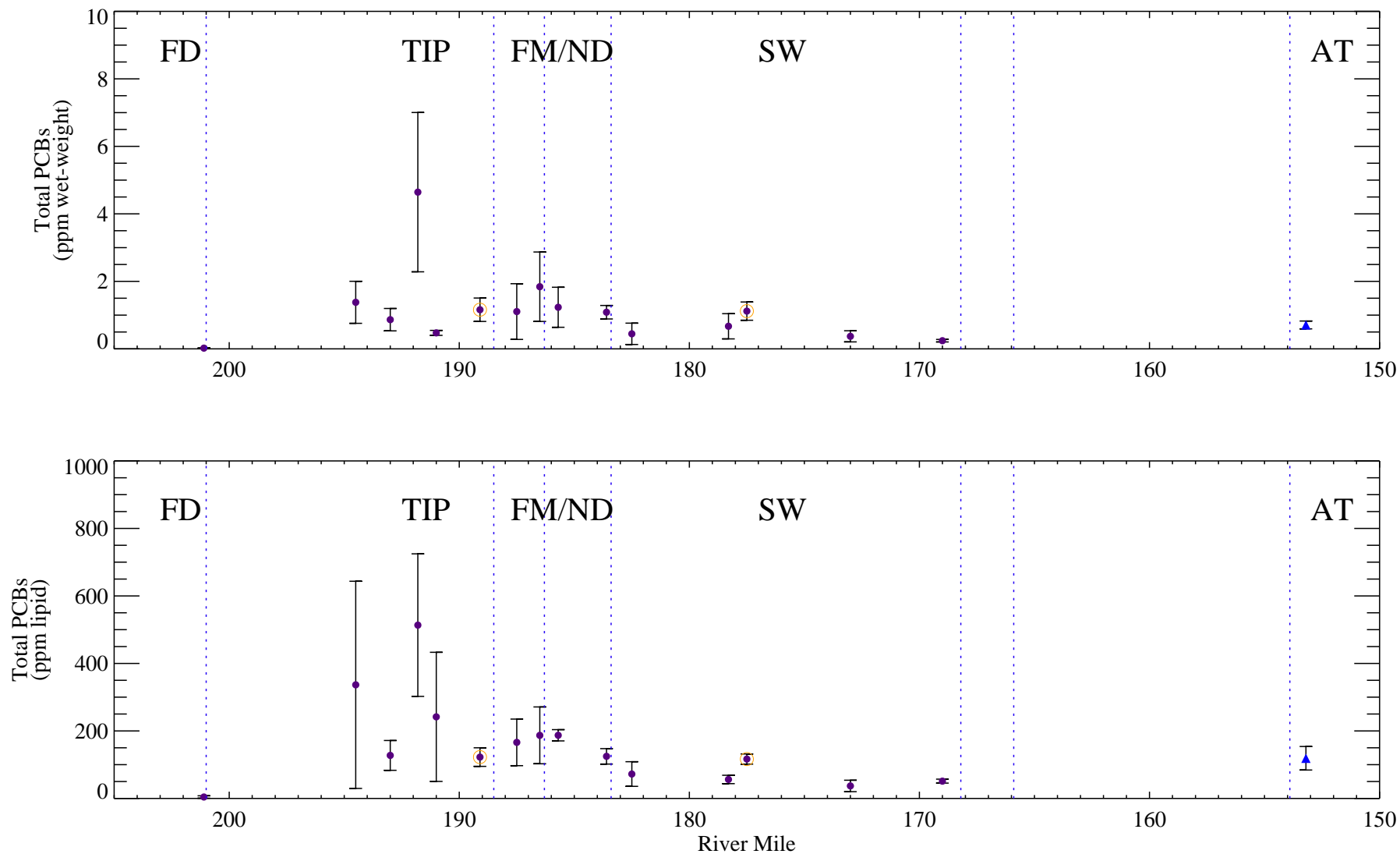
*Prep: fillet*

*Year: 2006.*

*Orange circles indicate historic sampling locations. Blue dotted lines indicate approximate dam locations.*

*Source: 2006 BMP (QEAExport\_Fish 03/08/2007).*





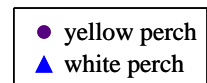
**Figure 5-4. Spatial patterns in PCB concentrations in perch.**

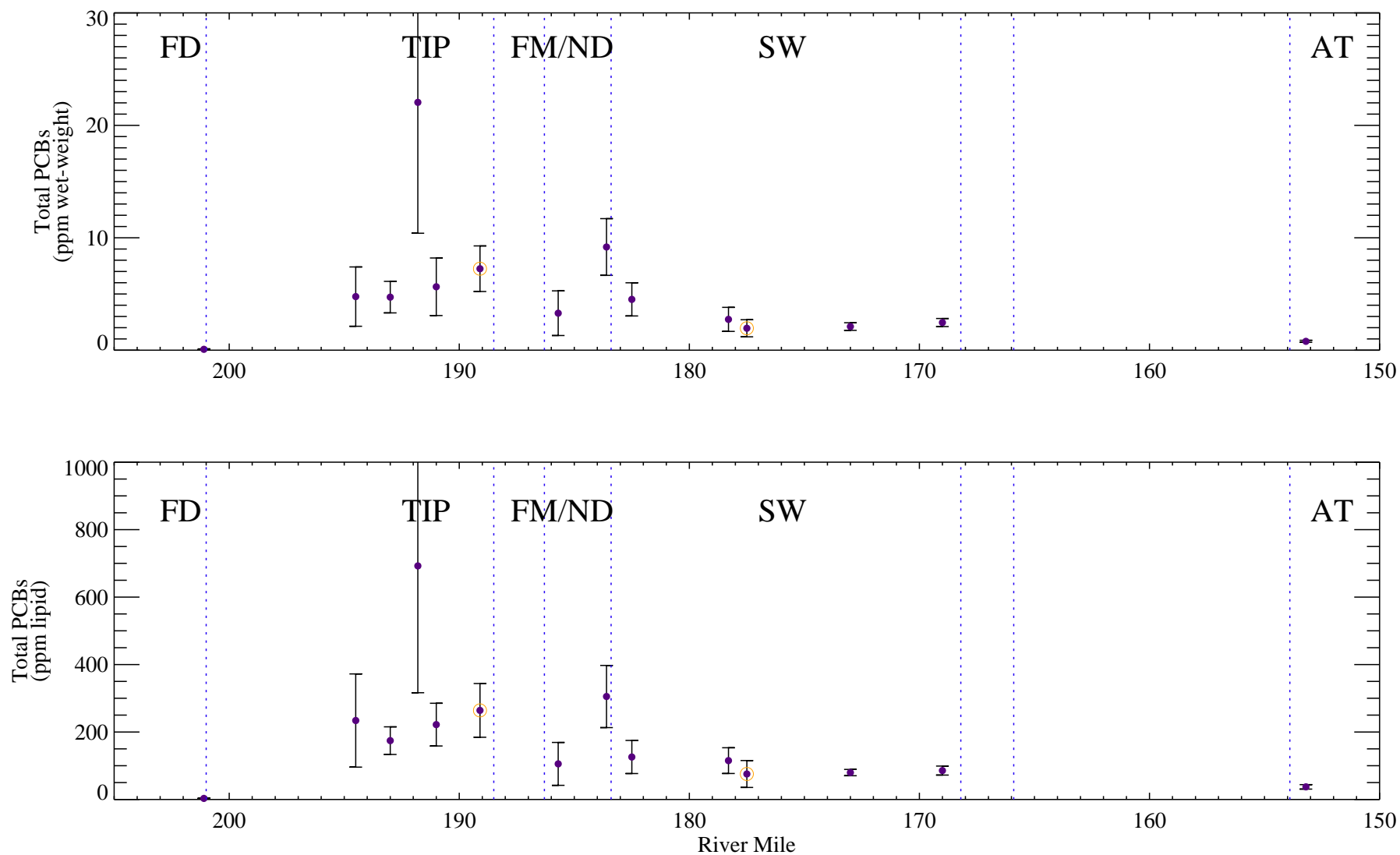
*Prep: fillet*

*Year: 2006.*

*Orange circles indicate historic sampling locations. Blue dotted lines indicate approximate dam locations.*

*Source: 2006 BMP (QEAExport\_Fish 03/08/2007).*





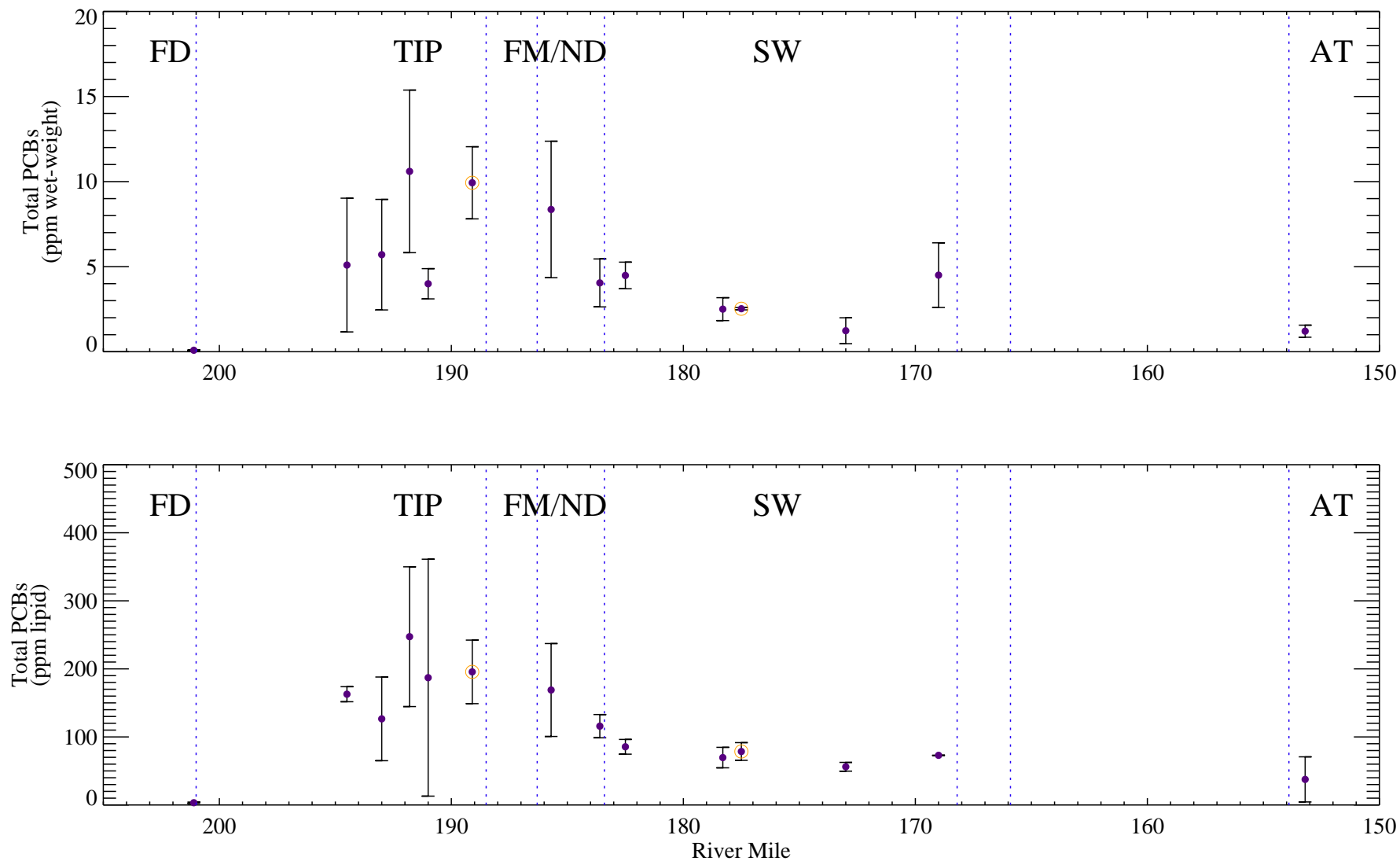
**Figure 5-5. Spatial patterns in PCB concentrations in pumpkinseed.**

*Prep: whole body*

*Year: 2006.*

*Orange circles indicate historic sampling locations. Blue dotted lines indicate approximate dam locations.*

*Source: 2006 BMP (QEAExport\_Fish 03/08/2007).*



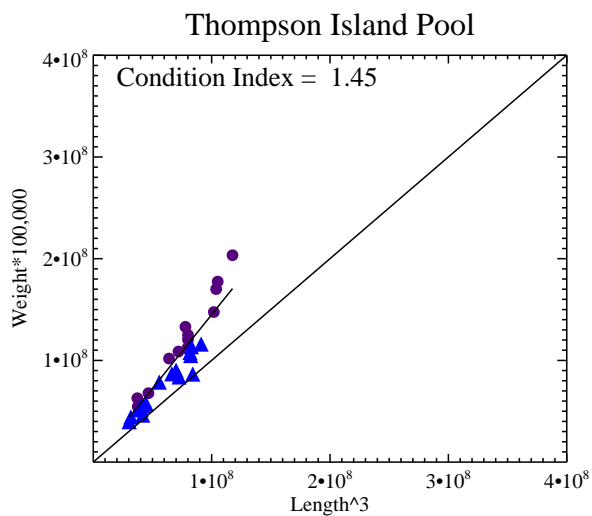
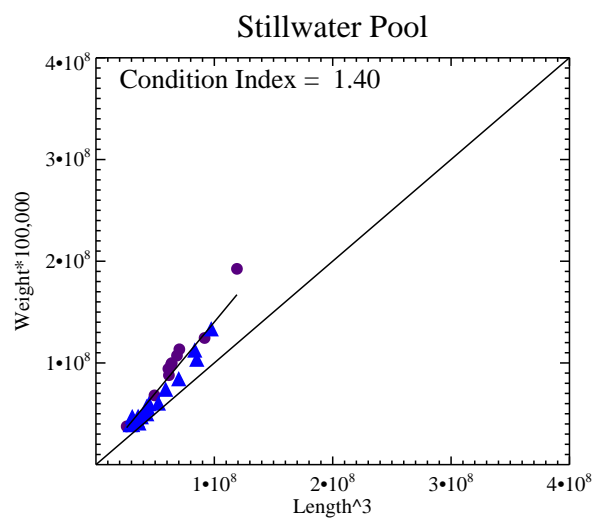
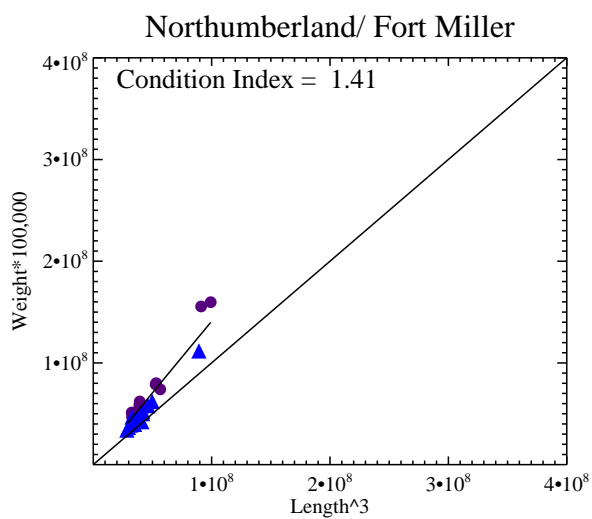
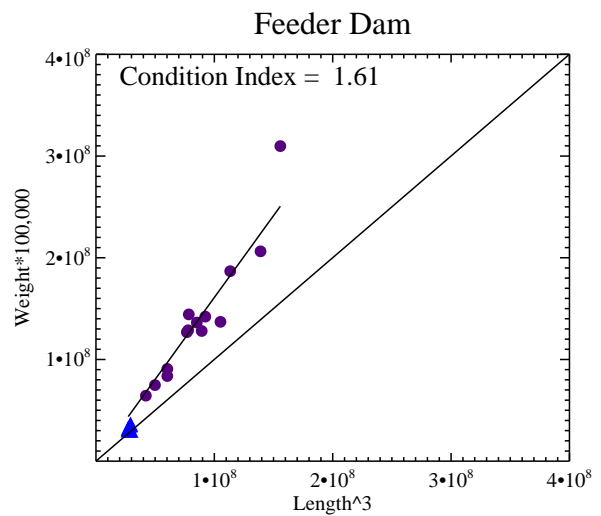
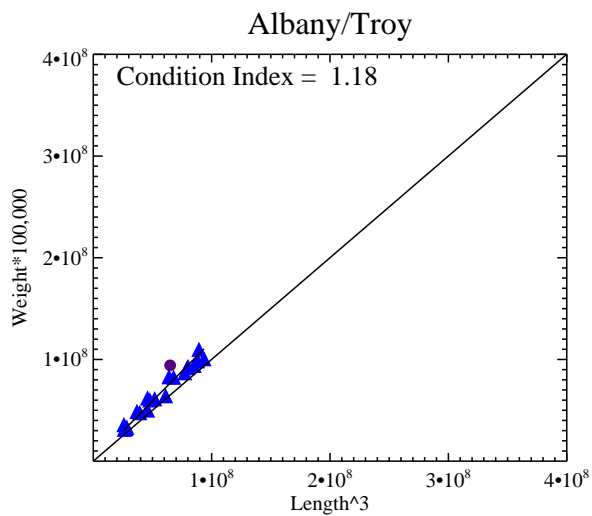
**Figure 5-6. Spatial patterns in PCB concentrations in forage fish.**

*Prep: whole-body composite*

*Year: 2006.*

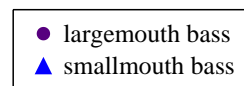
*Orange circles indicate historic sampling locations. Blue dotted lines indicate approximate dam locations.*

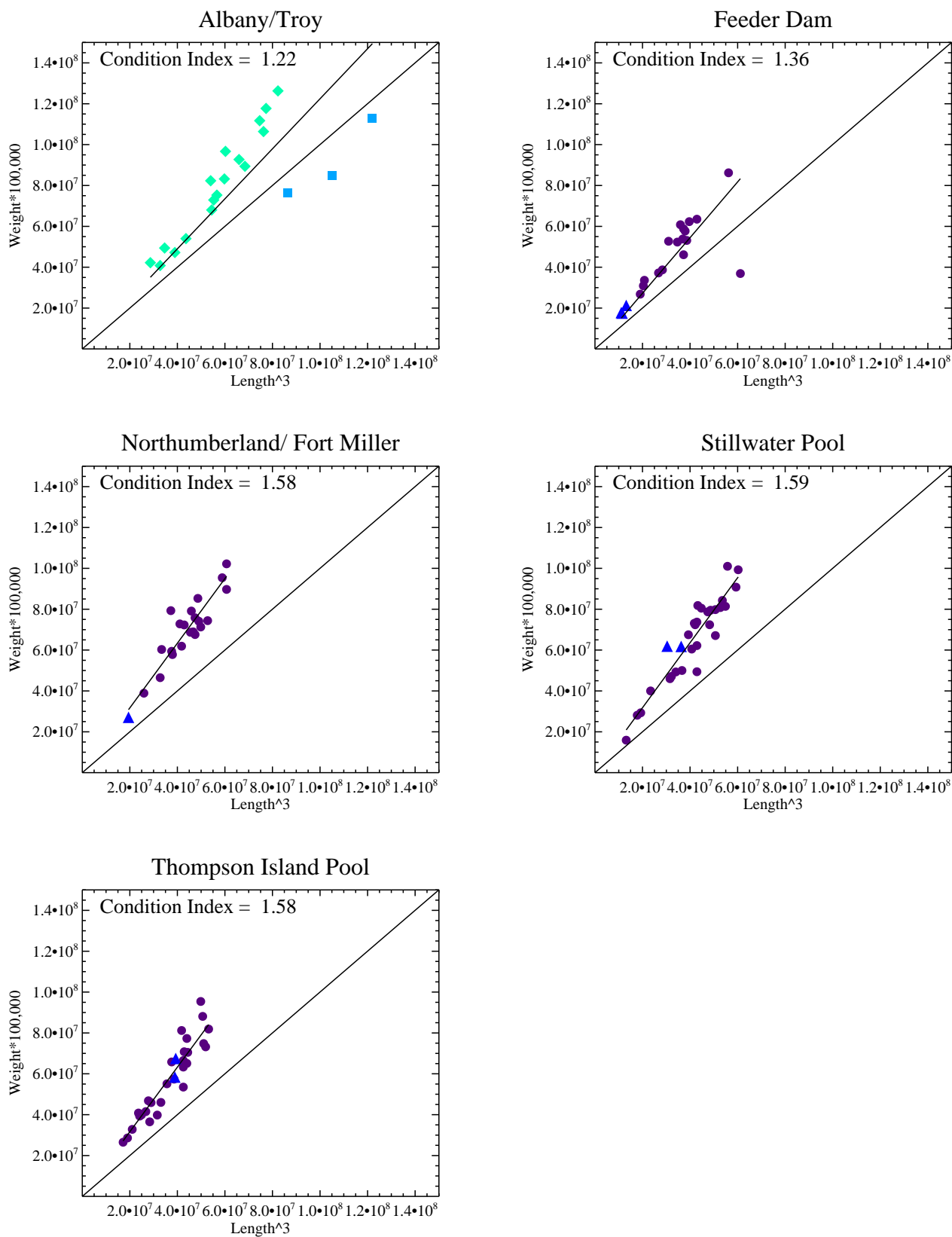
*Source: 2006 BMP (QEAExport\_Fish 03/08/2007).*



**Figure 5-7. Condition index of black bass.**

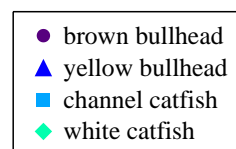
Spring and Autumn 2006 Sampling Events.  
Source: BMP (QEAExport\_Fish 03/08/2007).

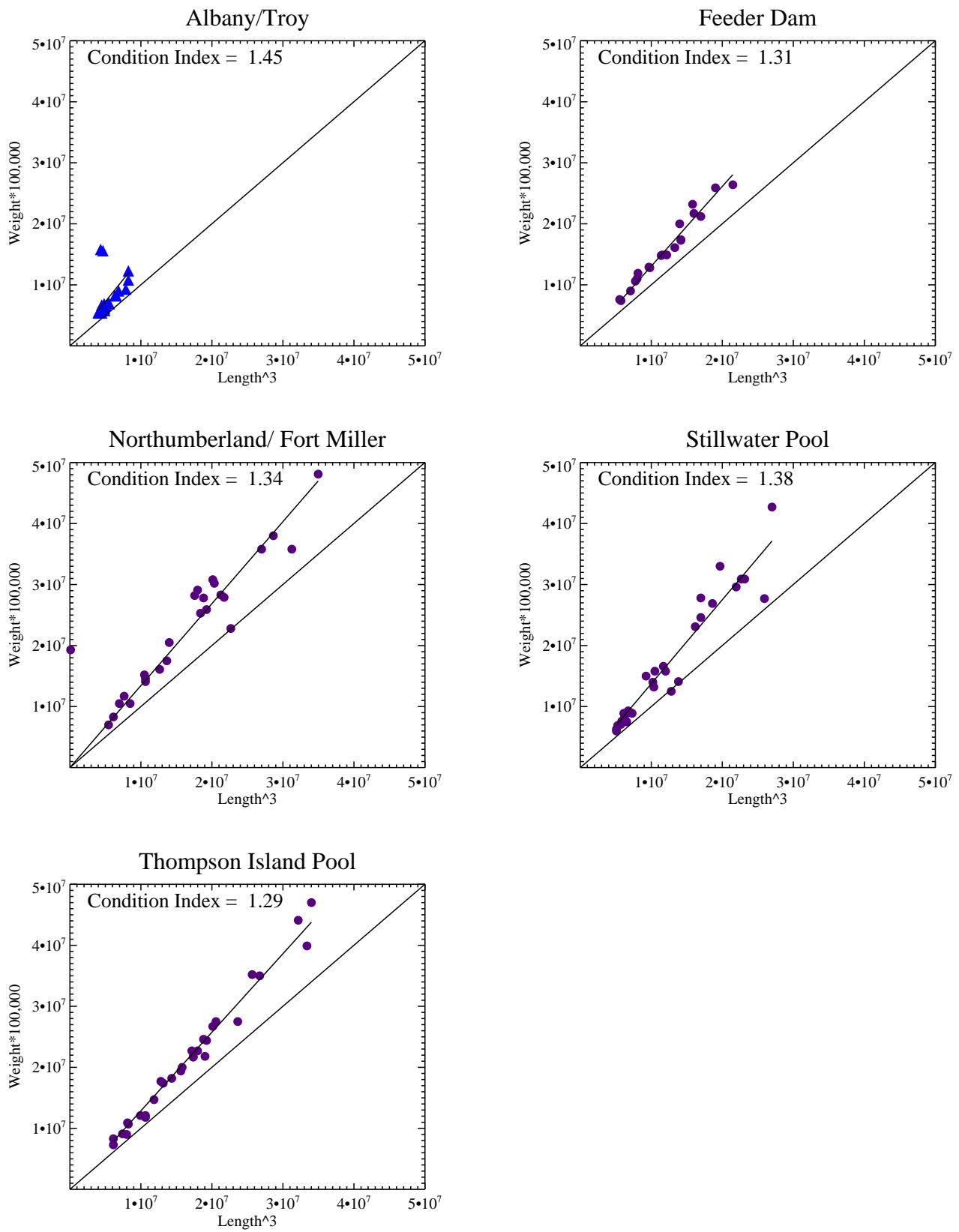




**Figure 5-8. Condition index of ictalurids.**

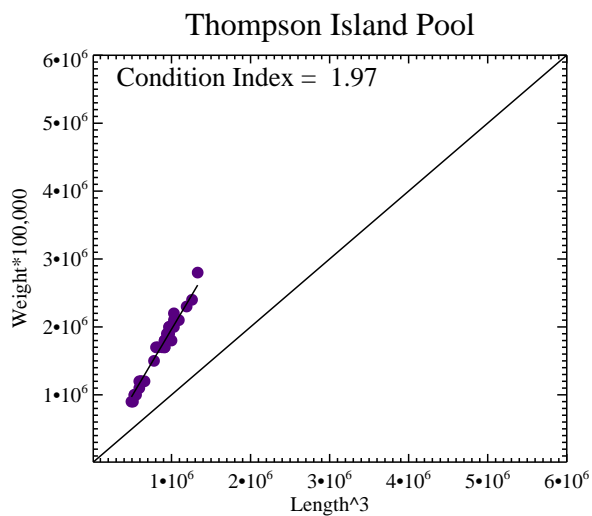
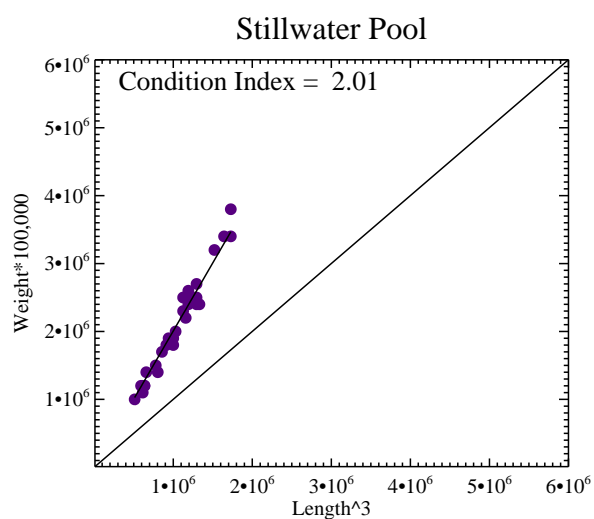
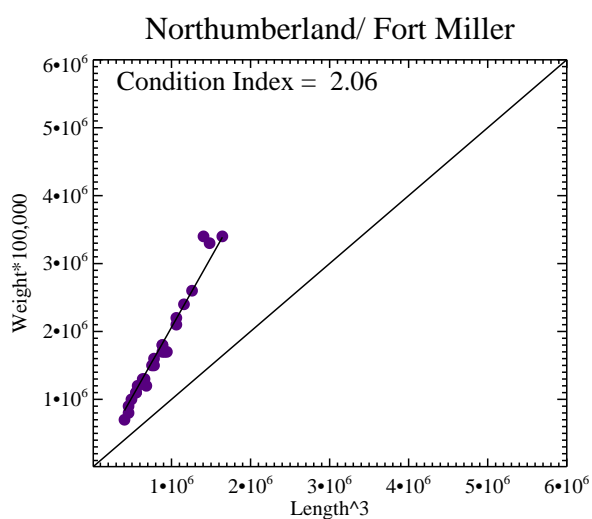
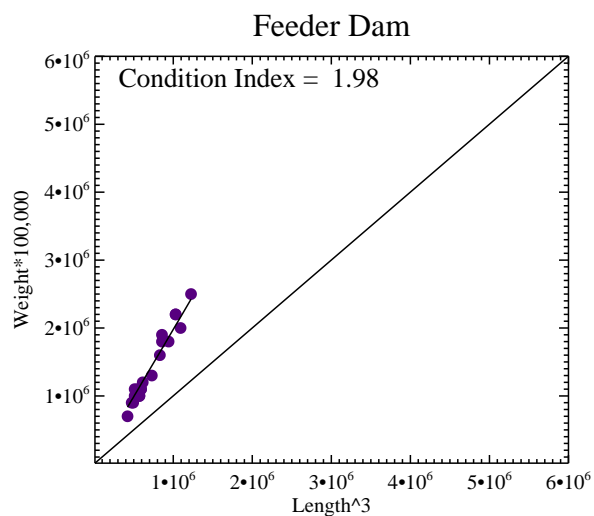
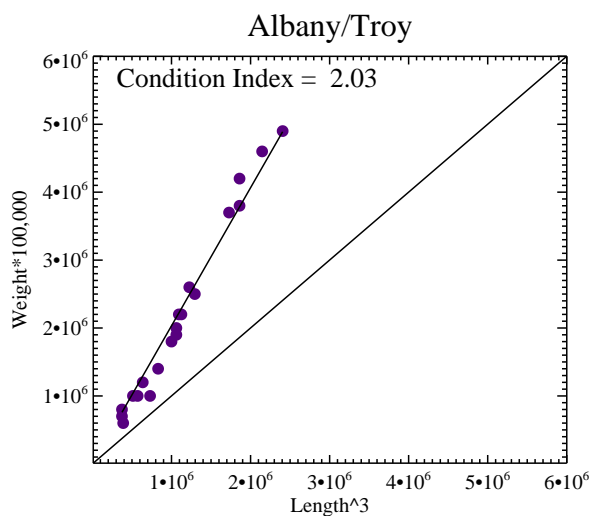
*Spring and Autumn 2006 Sampling Events.  
Source: BMP (QEAExport\_Fish 03/08/2007).*





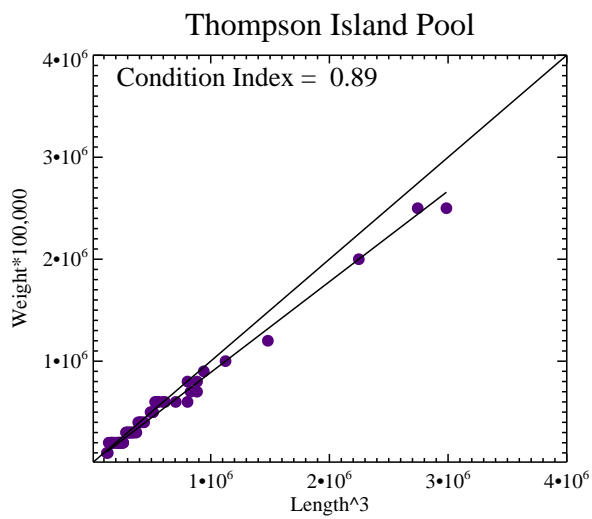
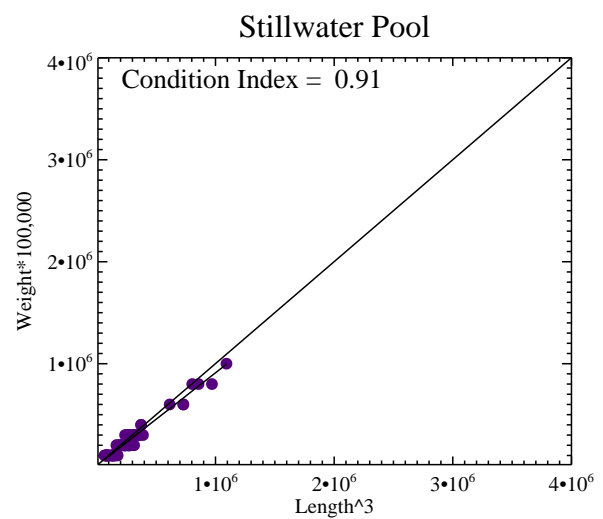
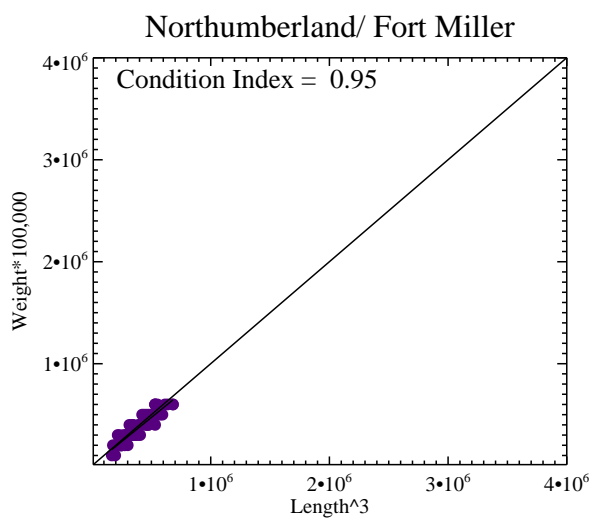
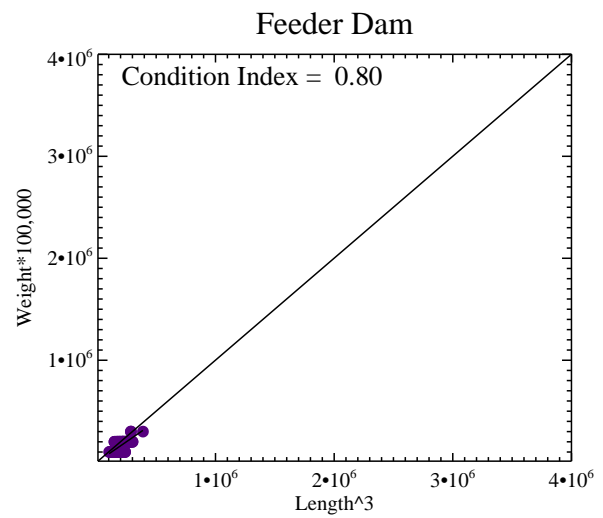
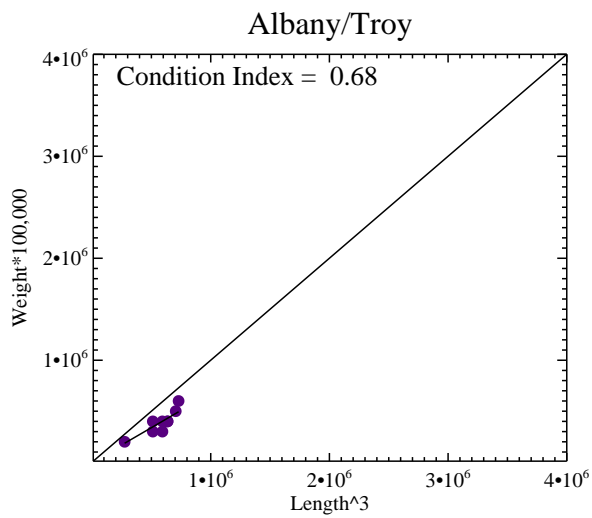
**Figure 5-9. Condition index of perch.**

Spring and Autumn 2006 Sampling Events.  
Source: BMP (QEAExport\_Fish 03/08/2007).



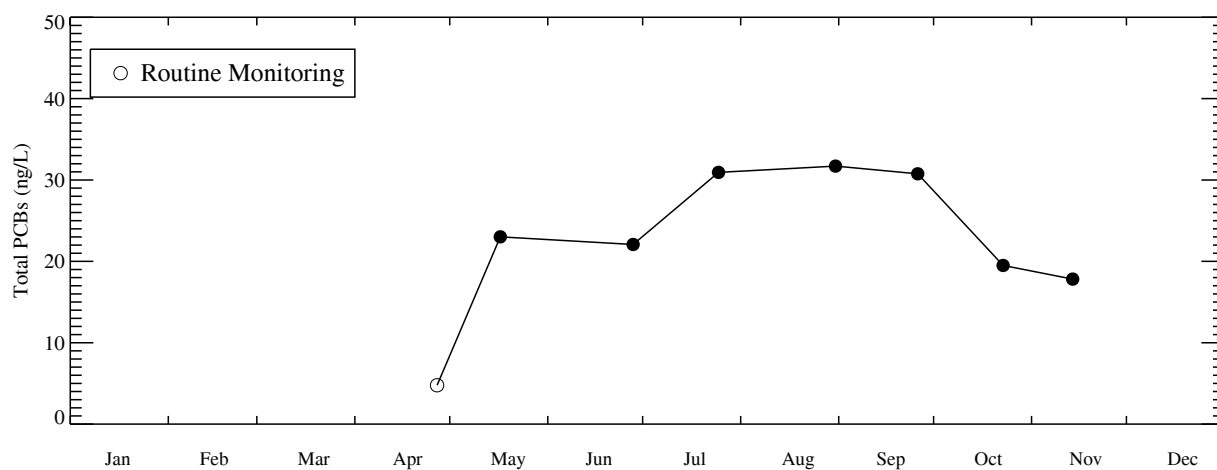
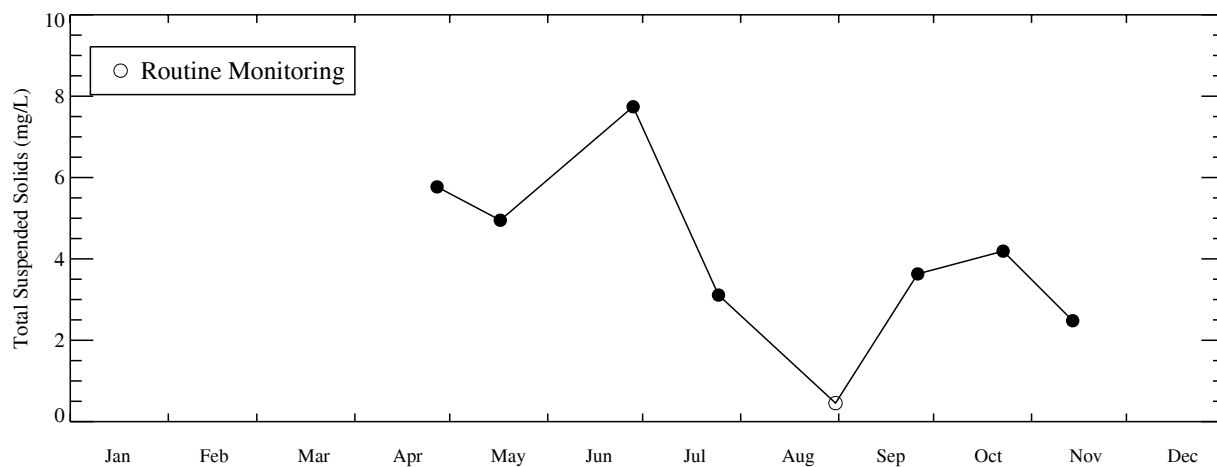
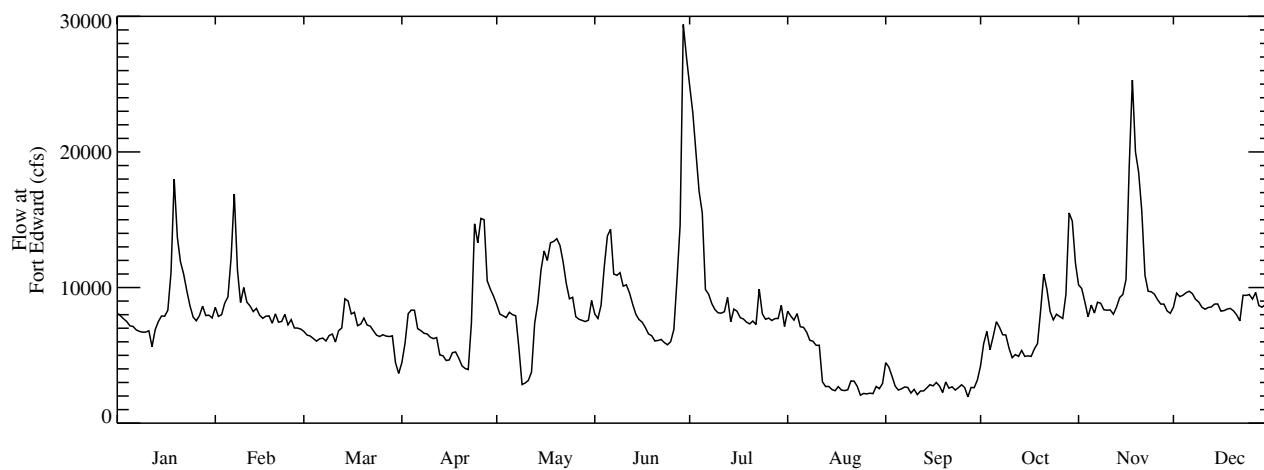
**Figure 5-10. Condition index of pumpkinseed.**

*Spring and Autumn 2006 Sampling Events.  
Source: BMP (QEAExport\_Fish 03/08/2007).*



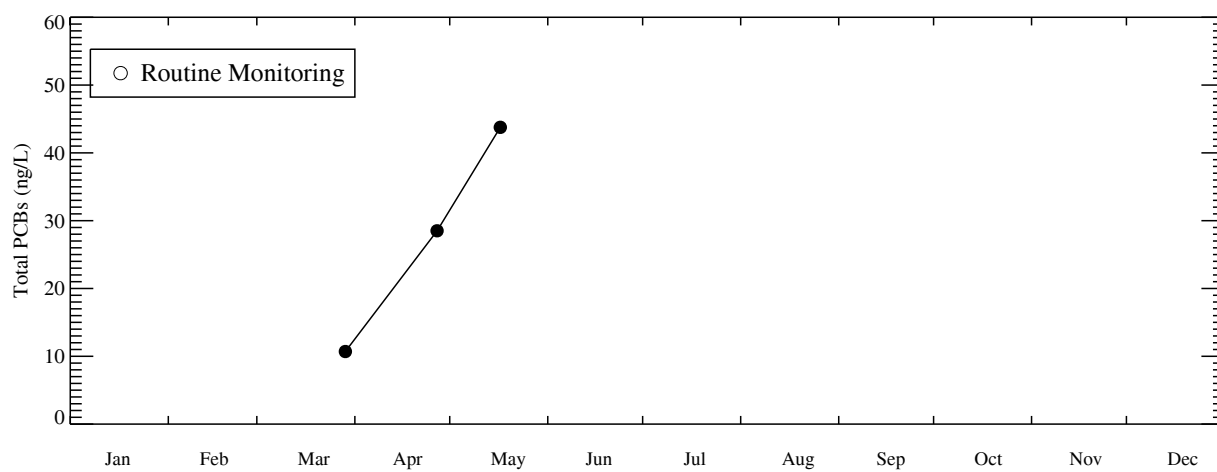
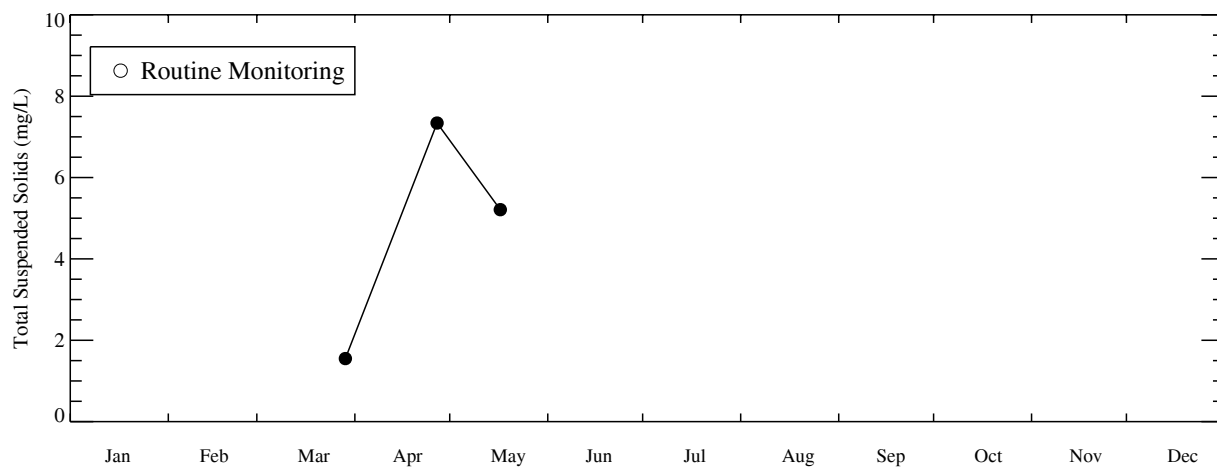
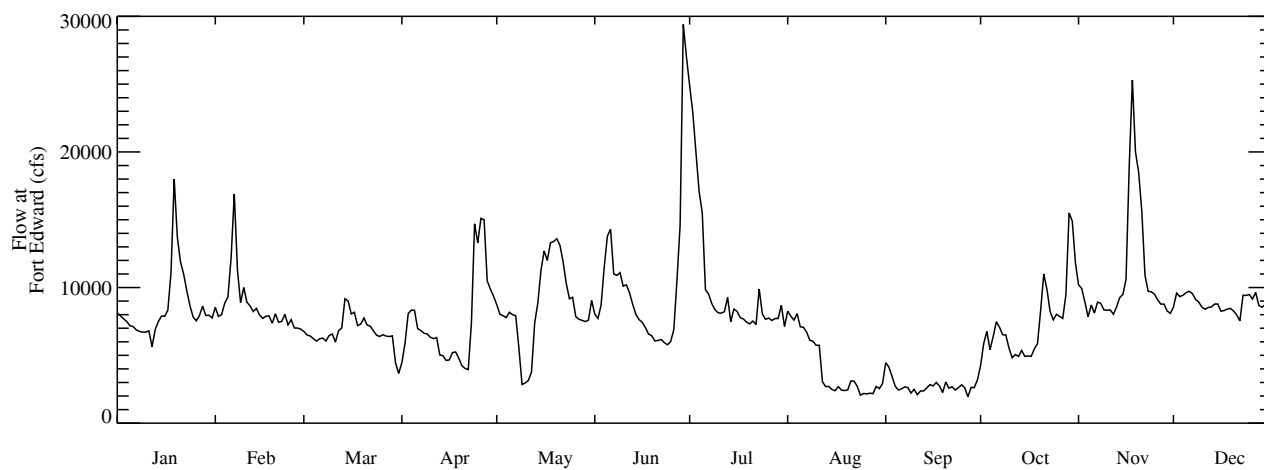
**Figure 5-11. Condition index of forage fish.**

*Spring and Autumn 2006 Sampling Events.  
Source: BMP (QEAExport\_Fish 03/08/2007).*



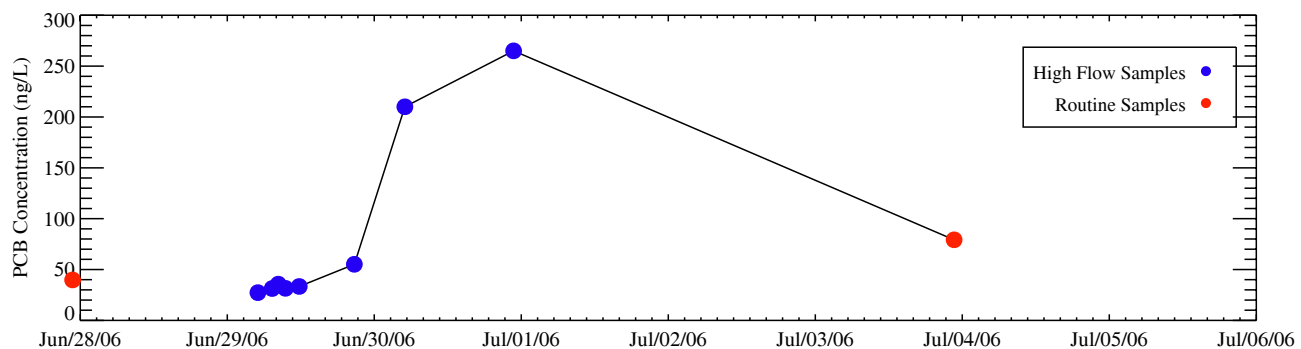
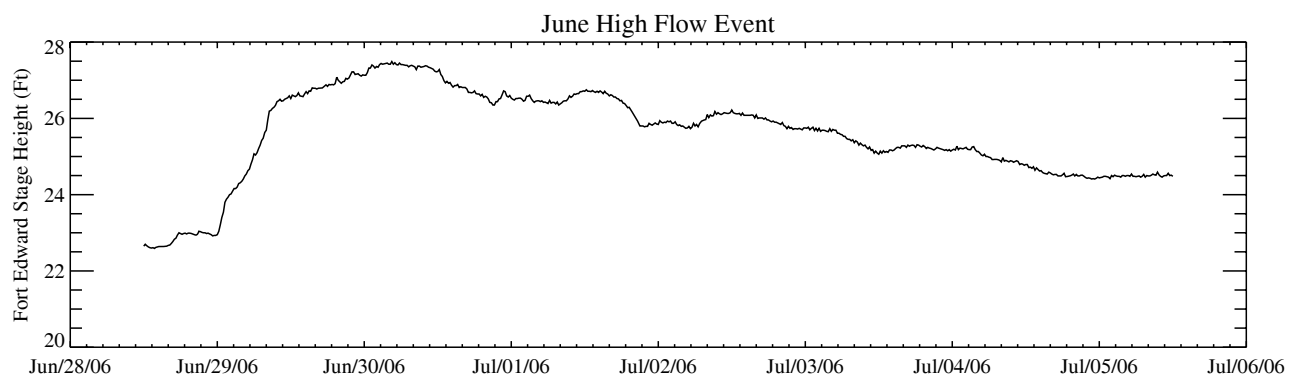
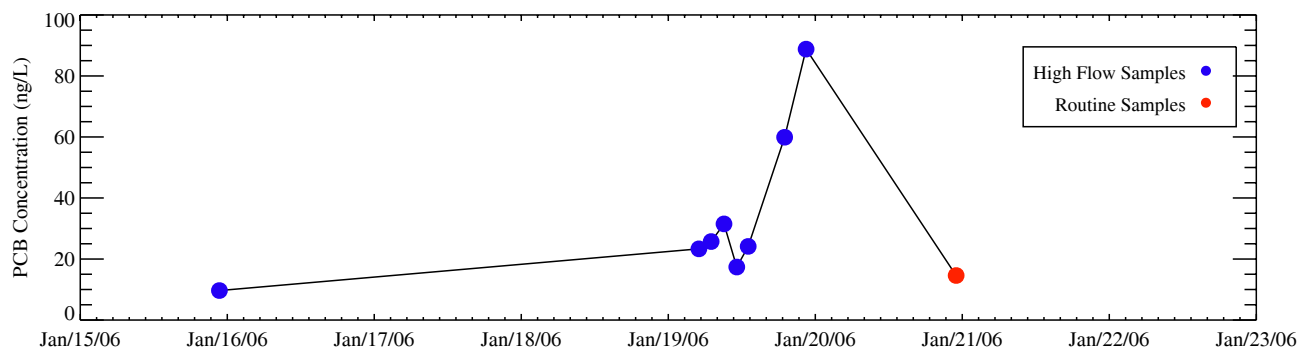
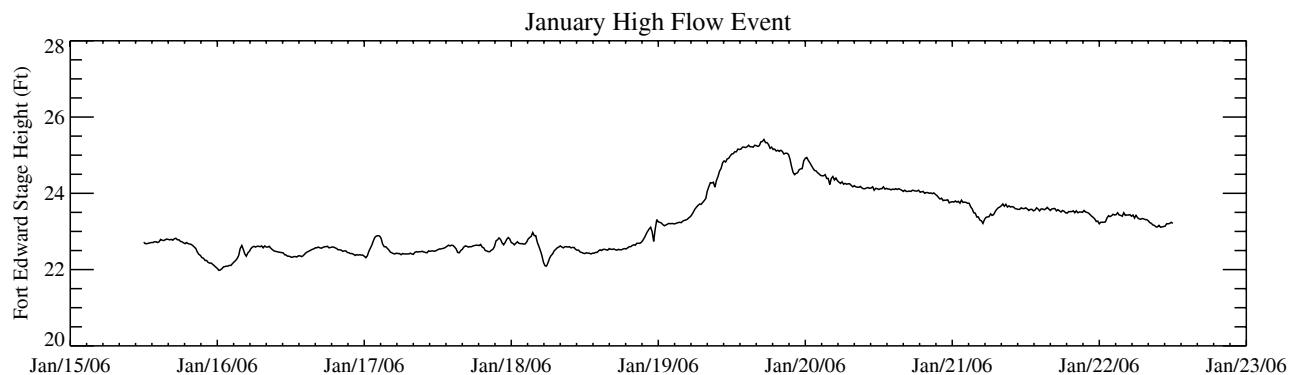
**Figure 6-1. 2006 Temporal profiles of PCB and TSS results at TID-PRW2.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



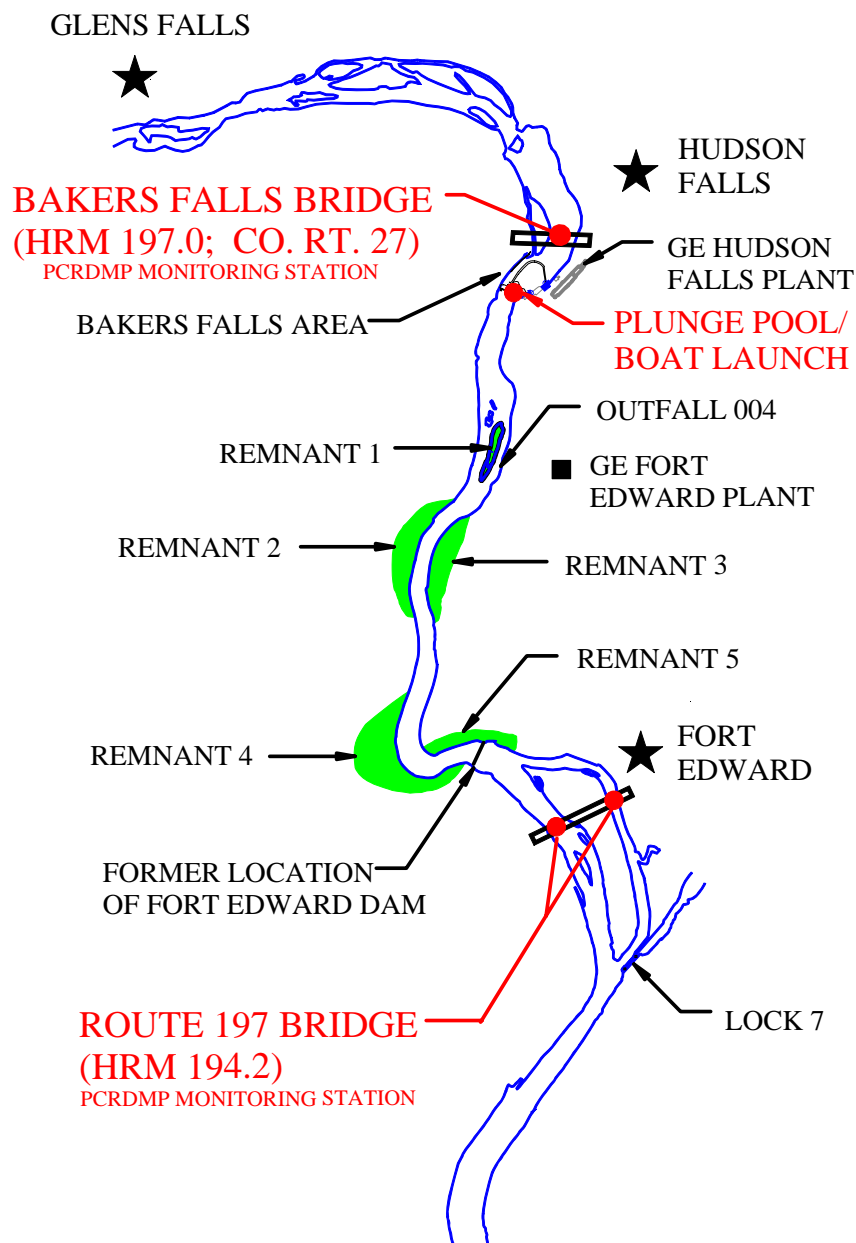
**Figure 6-2. 2006 Temporal profiles of PCB and TSS results at Schuylerville Center.**

*Non-detects plotted at half the detection limit with open symbols. Samples not plotted on the line are blind duplicates.*



**Figure 6-3. Waterford High Flow Sampling Events.**

- Notes:
- USGS 15 minute stage data is provisional and subject to revision.
  - Plots include high flow events with 6 or more sampling rounds.



# **LEGEND**

● - WATER SAMPLING LOCATION

GENERAL ELECTRIC COMPANY  
POST CONSTRUCTION REMNANT DEPOSIT  
MONITORING PROGRAM  
SAMPLE LOCATION MAP

FIGURE 7-1

SCALE: 1 in. = 4000 ft.

FINAL



GENbmp 131

MARCH 2007

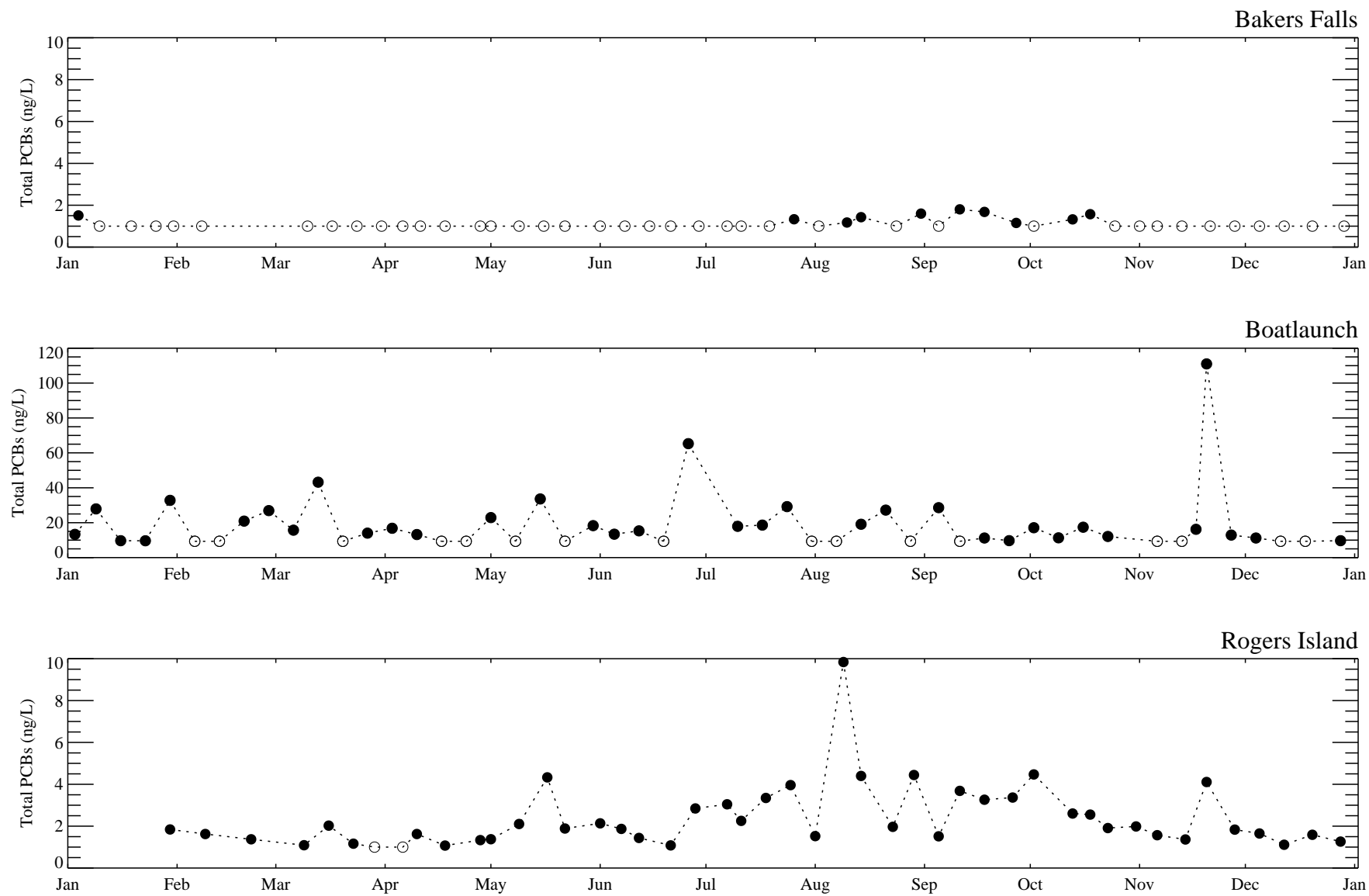


Figure 7-2. Temporal profiles of 2006 routine monitoring data collected in the vicinity of Hudson Falls.

Notes: Blind duplicate samples averaged. Non-detect total PCB samples set to the MDL (1.1 ng/L).

## APPENDICES

**APPENDIX A**  
**CORRECTIVE ACTION MEMORANDA**  
**(CD-ROM ATTACHED)**

**GENERAL ELECTRIC COMPANY  
HUDSON RIVER BASELINE MONITORING PROGRAM  
CORRECTIVE ACTION MEMORANDUM NO. 5**

**Date:** April 27, 2006

**Organization Name:** Quantitative Environmental Analysis, LLC

**Initiator's Name and Title:** Christopher Yates

**Problem Description:**

The Baseline Monitoring Program (BMP) Quality Assurance Project Plan (QAPP; QEA and ESI 2004) identifies data quality objectives (DQOs) that were to be met through the collection of certain data in 2004 during the seven month period (May through November) that coincides with the anticipated construction season for remedial dredging. One of these DQOs required the collection and analysis of samples on a weekly basis to establish baseline concentrations of nutrients (total Kjeldahl nitrogen [TKN], nitrite, nitrate, and total phosphorous) prior to dredging. As the BMP was not started until June 2004, no nutrient data were collected in May 2004. To fill this data gap, nutrient data were collected again in 2005 (May – November).

Nutrients were monitored weekly from June through November 2004 and May through November 2005 at all Upper Hudson River stations. Spatial trends for TKN, nitrite, nitrate, and phosphorus are presented in Figures 1a – 1d, respectively, for each weekly sampling event. Figures 2a – 2d present temporal trends for the same parameter. Nearly all TKN concentrations are within the range of approximately 0.3 to 0.6 ug/L; significant spatial or temporal trends are not evident. With the exception of a few data points, nitrite concentrations range from <0.01 to approximately 0.04 mg/L and nitrate concentrations range from approximately 0.2 to 0.6 mg/L. Similar to TKN, significant spatial or temporal trends are not evident in either the nitrite or nitrate data. Total Phosphorus concentrations were largely at or below the method detection limit of 50 µg/L; therefore, no spatial or temporal trends can be identified. These results indicate minimal variability in the nutrient data; the BMP QAPP DQO of documenting baseline concentrations have been satisfied with current data.

**Reported To:** Bob Gibson, GE; John Haggard, GE; John Connolly, QEA

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**Corrective Action:**

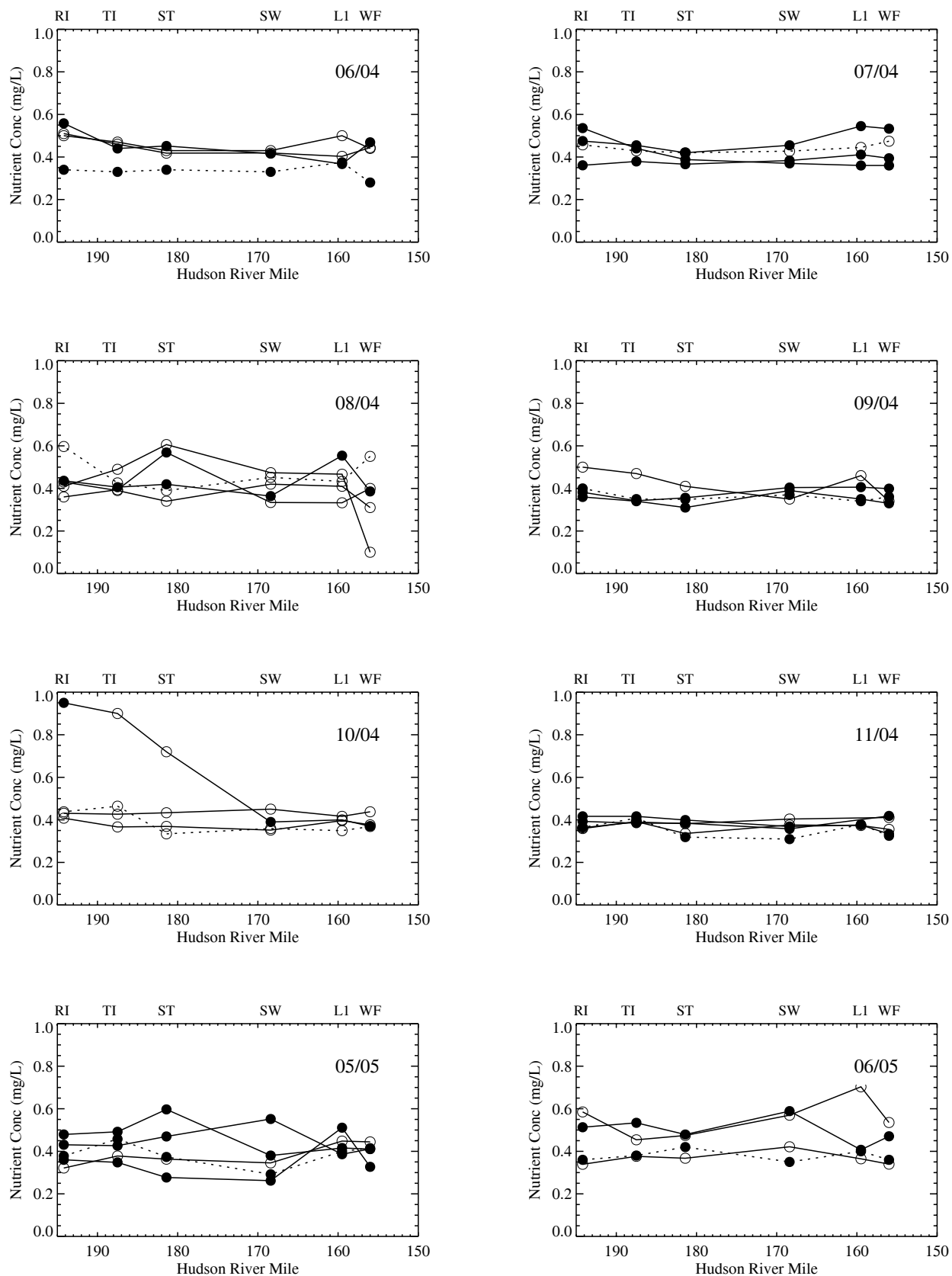
Effective May 1, 2006, GE will discontinue collecting samples for nutrient analysis at all stations in the baseline monitoring program.

**Reviewed and Implemented By:** Christopher Yates (QEA)

cc: GE Program Manager: John Haggard; Bob Gibson

Field Program Manager: Mark LaRue (QEA)

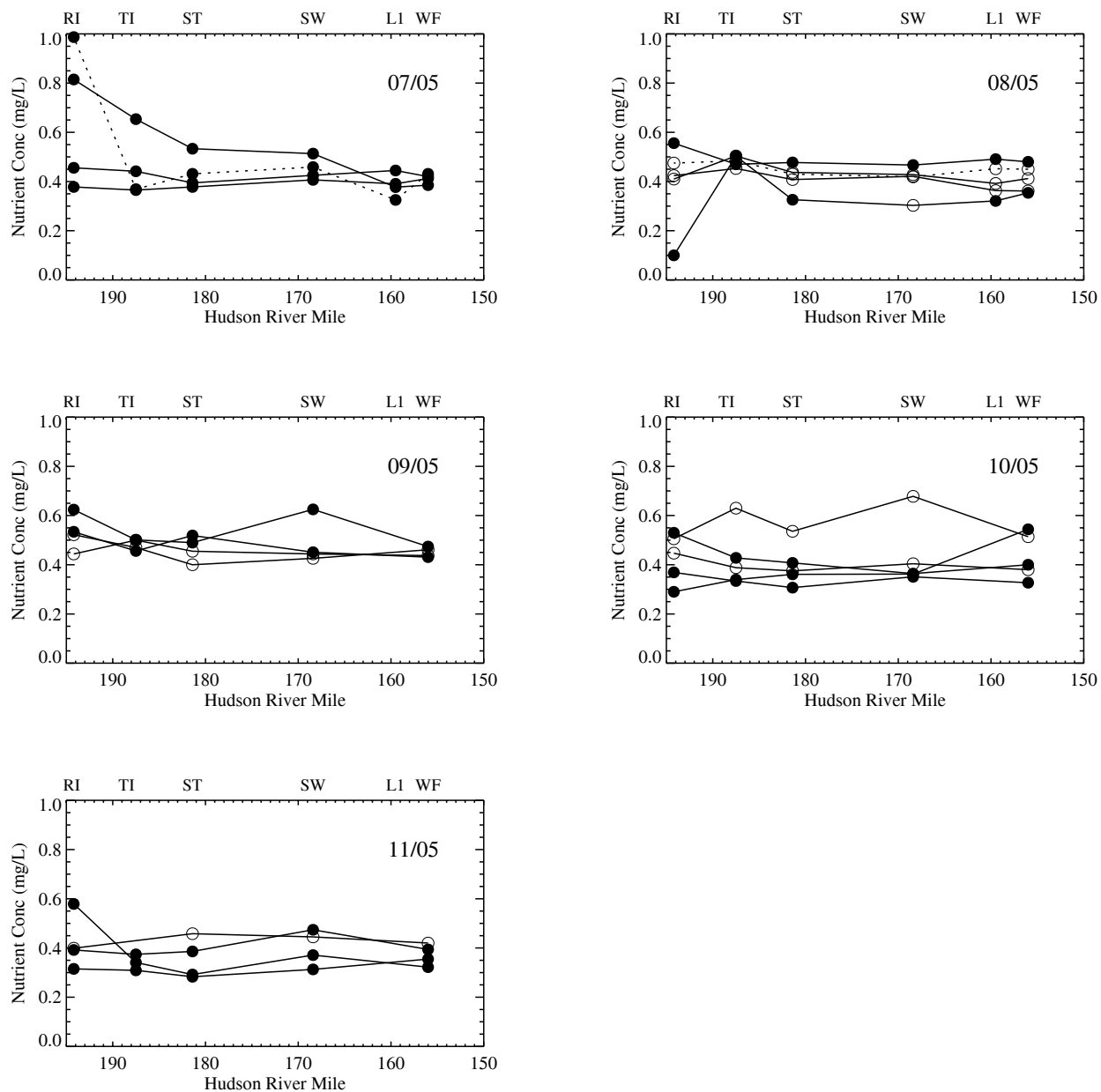
Other Distribution: John Connolly (QEA), Jim Rhea (QEA), Laurie Scheuing (QEA)  
David Blye (ESI); Bob Wagner (NEA)



**Figure 1a. Spatial trends in Upper Hudson River TKN concentrations.**

*Notes: Solid line indicates a routine sample event, dotted line indicates a Time of Travel sampling event.*

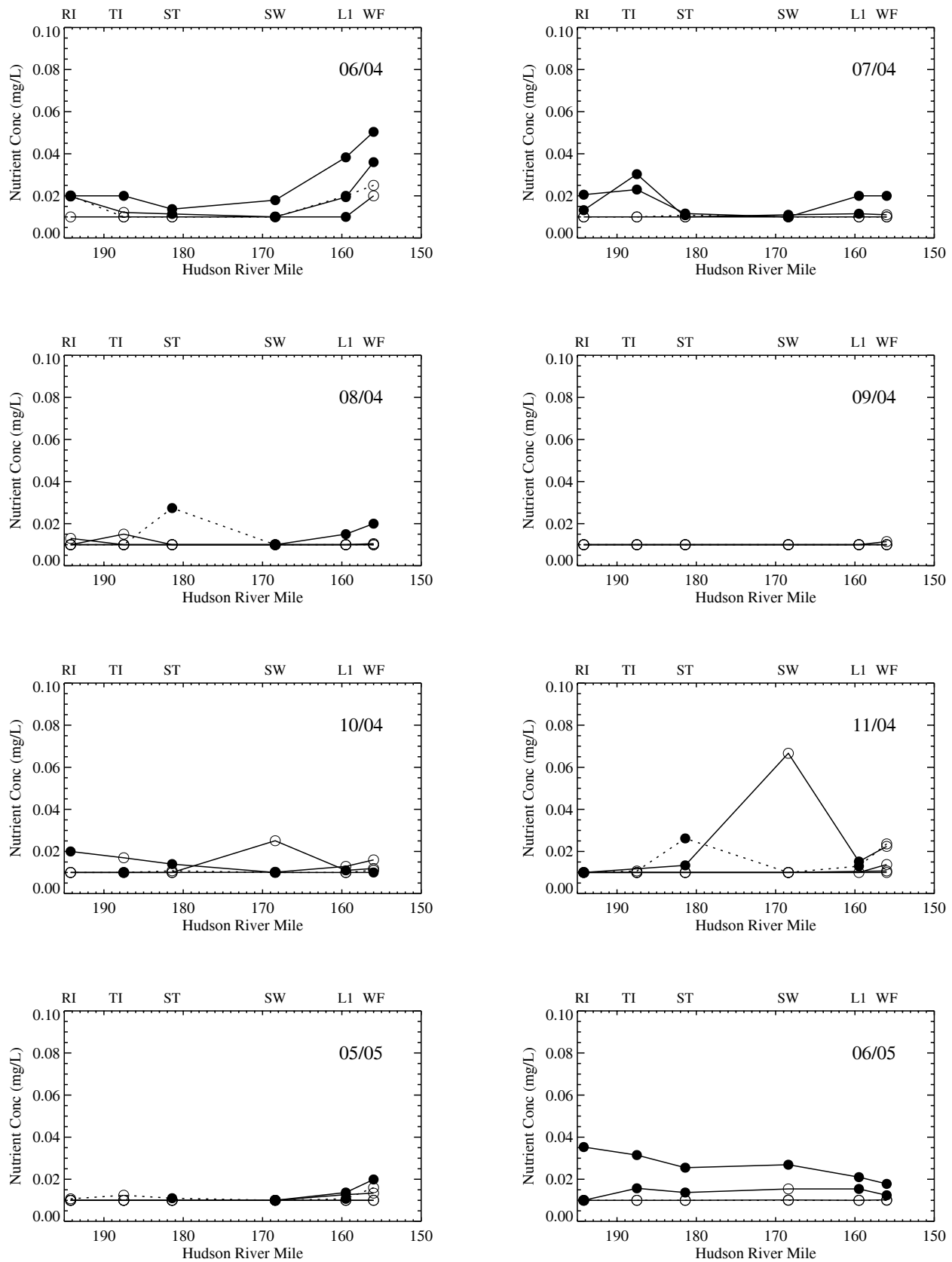
*Non-detect samples set to the MDL and plotted with open symbols.*



**Figure 1a. Spatial trends in Upper Hudson River TKN concentrations.**

*Notes: Solid line indicates a routine sample event, dotted line indicates a Time of Travel sampling event.*

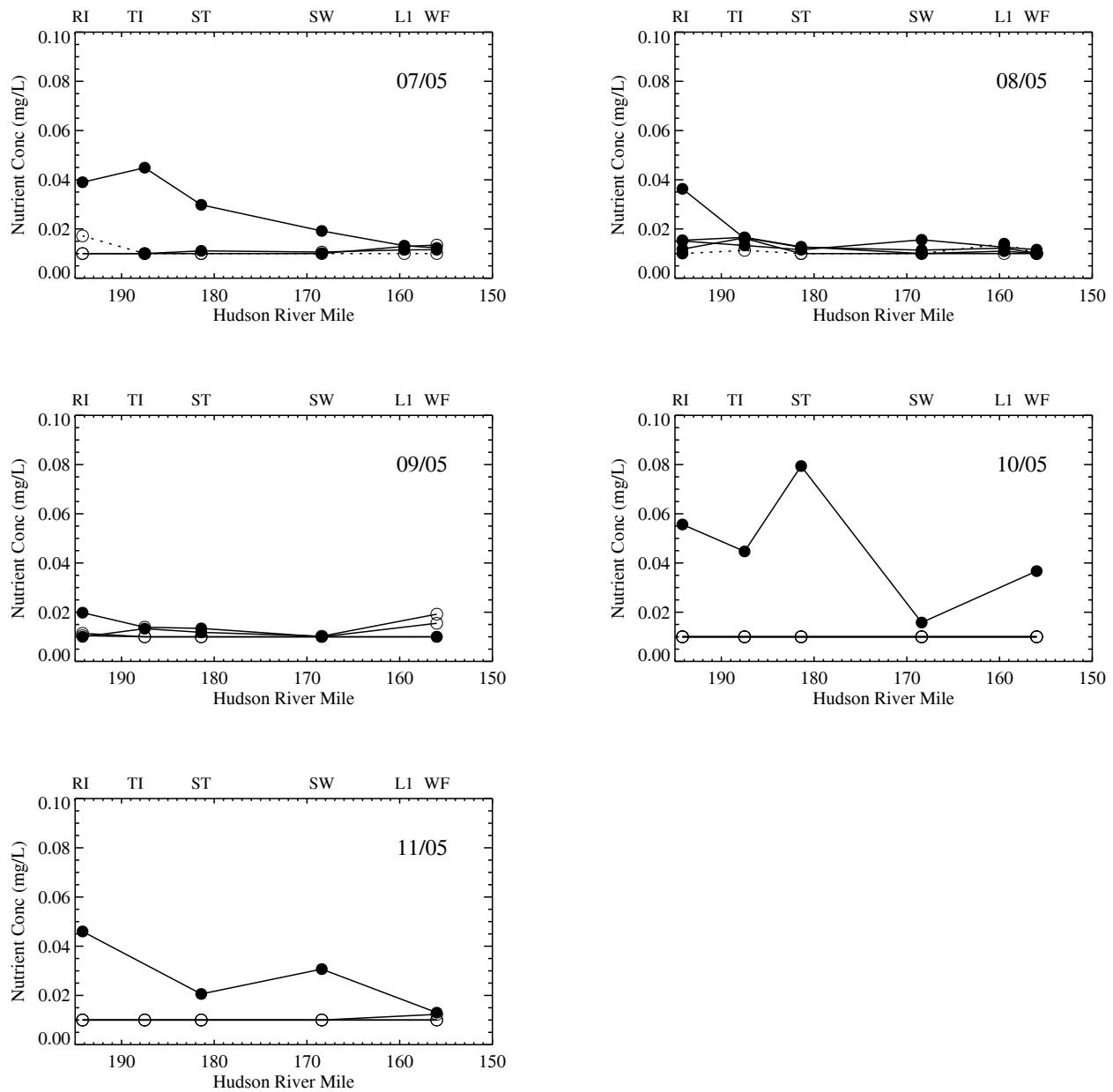
*Non-detect samples set to the MDL and plotted with open symbols.*



**Figure 1b. Spatial trends in Upper Hudson River Nitrite concentrations.**

*Notes: Solid line indicates a routine sample event, dotted line indicates a Time of Travel sampling event.*

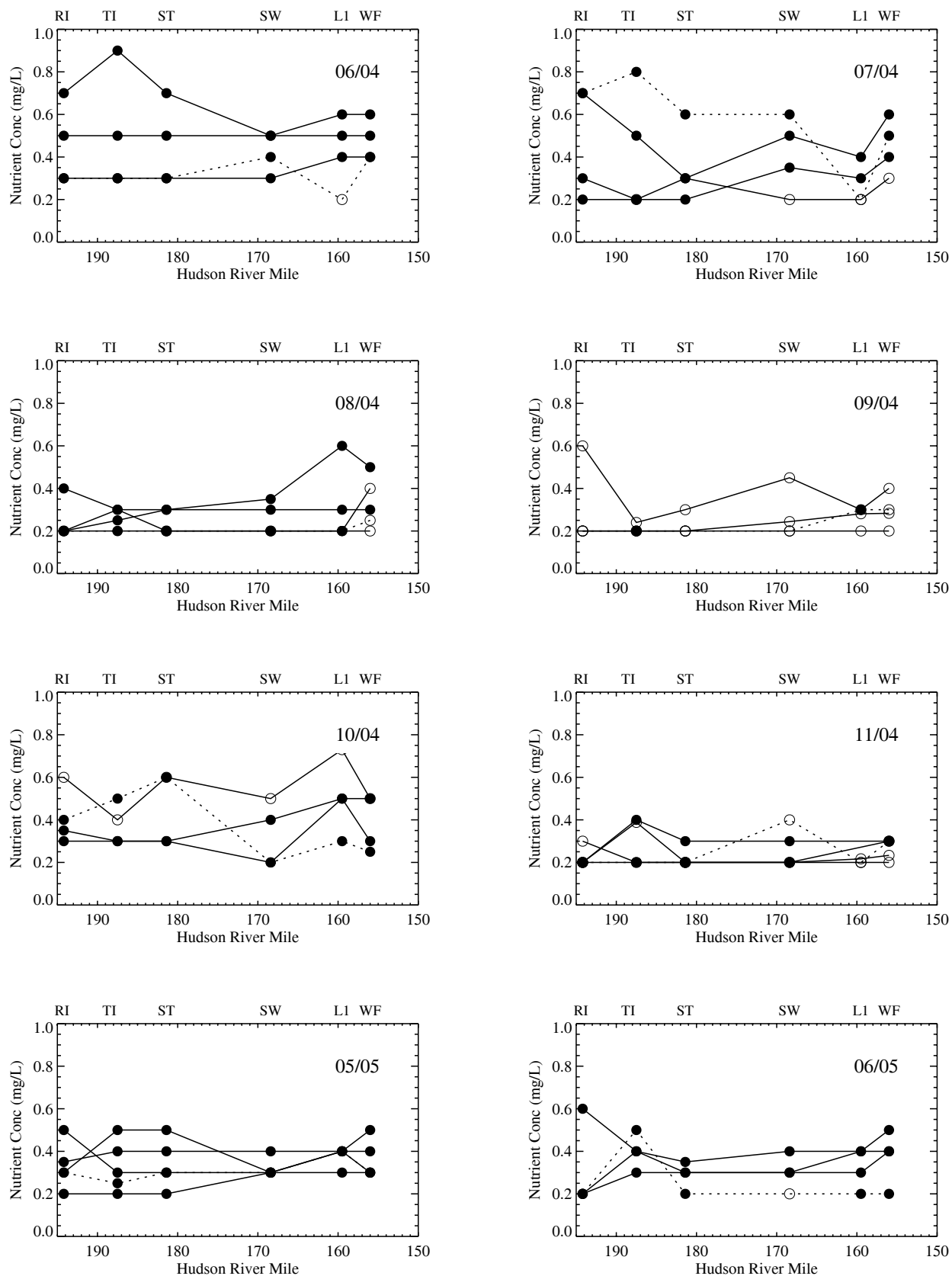
*Non-detect samples set to the MDL and plotted with open symbols.*



**Figure 1b. Spatial trends in Upper Hudson River Nitrite concentrations.**

*Notes: Solid line indicates a routine sample event, dotted line indicates a Time of Travel sampling event.*

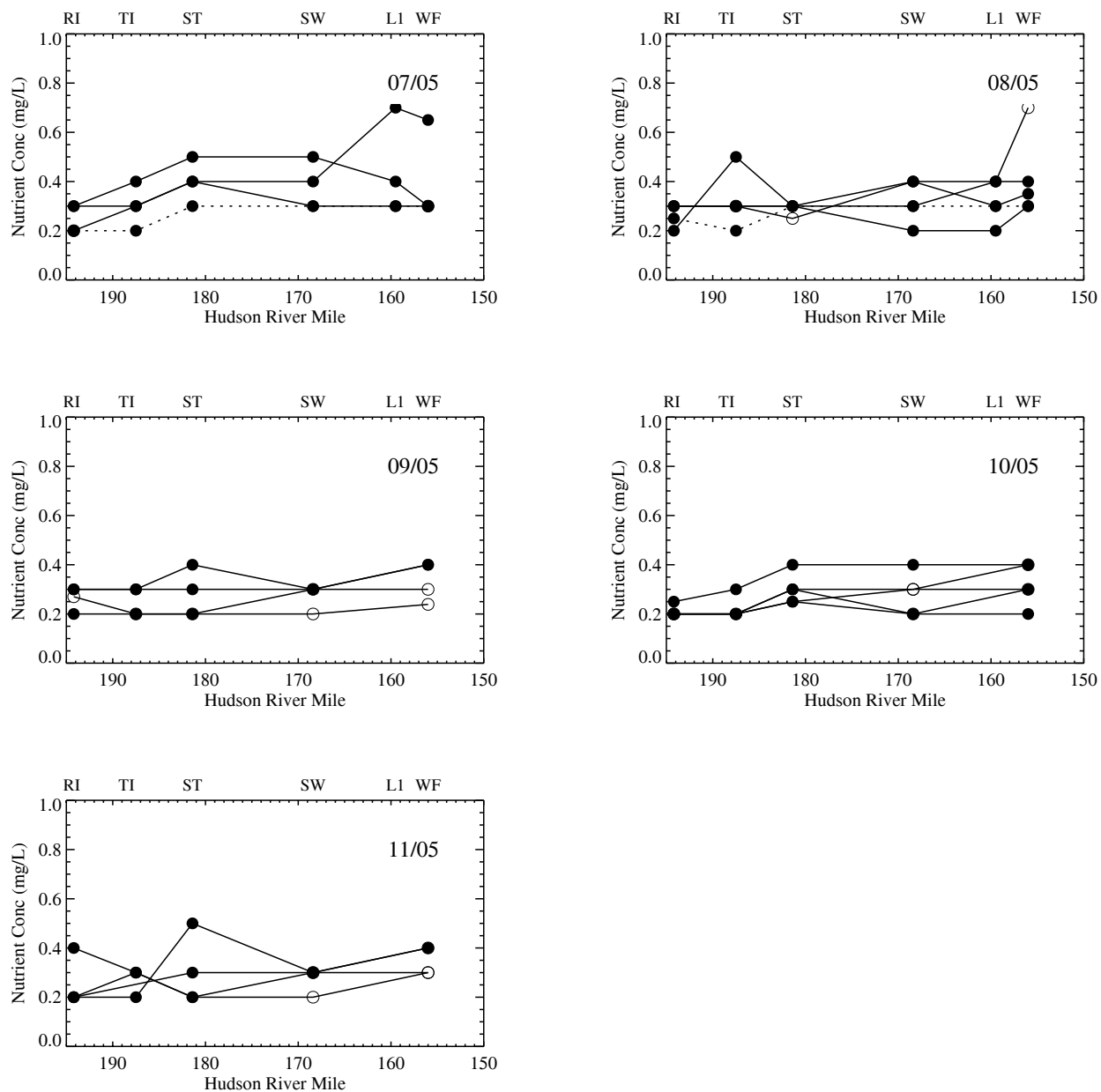
*Non-detect samples set to the MDL and plotted with open symbols.*



**Figure 1c. Spatial trends in Upper Hudson River Nitrate concentrations.**

*Notes: Solid line indicates a routine sample event, dotted line indicates a Time of Travel sampling event.*

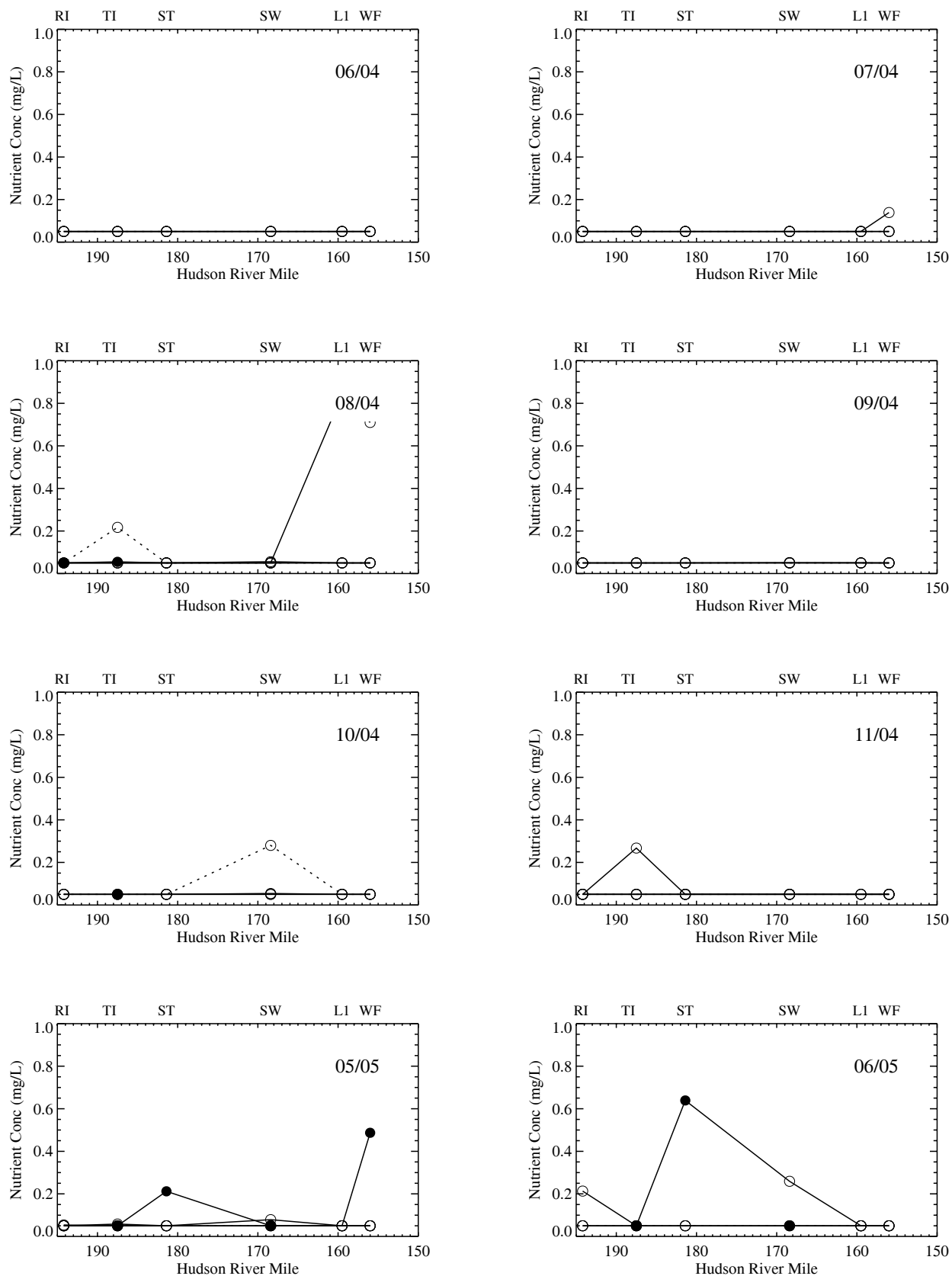
*Non-detect samples set to the MDL and plotted with open symbols.*



**Figure 1c. Spatial trends in Upper Hudson River Nitrate concentrations.**

*Notes: Solid line indicates a routine sample event, dotted line indicates a Time of Travel sampling event.*

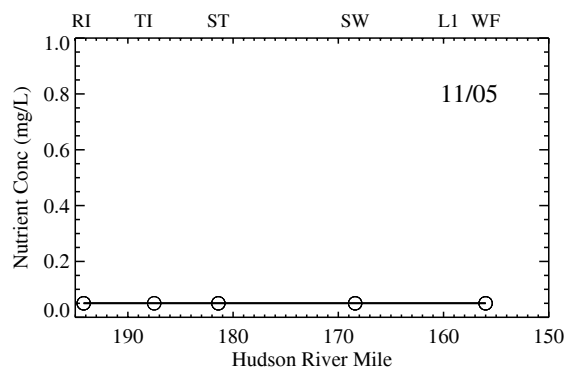
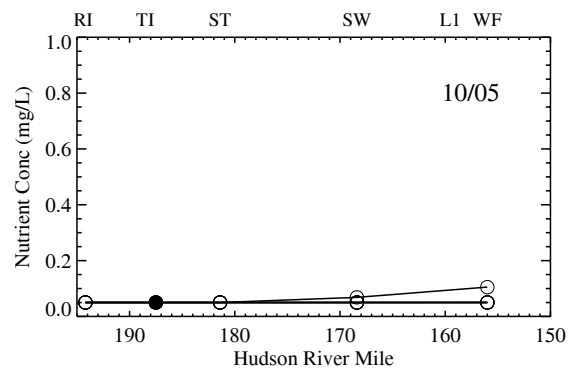
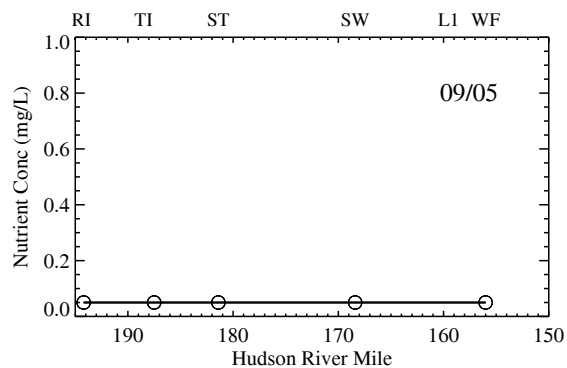
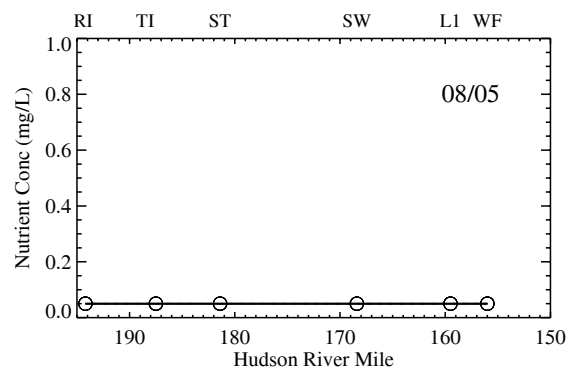
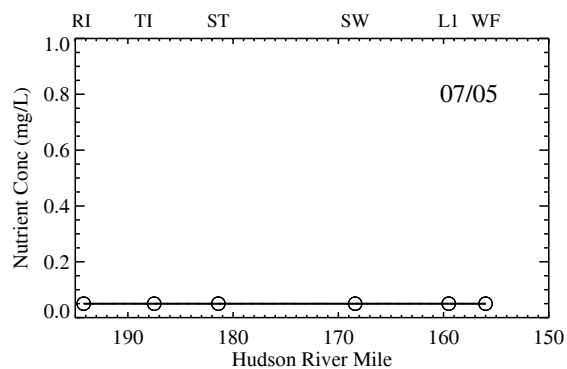
*Non-detect samples set to the MDL and plotted with open symbols.*



**Figure 1d. Spatial trends in Upper Hudson River Total Phosphorous concentrations.**

*Notes: Solid line indicates a routine sample event, dotted line indicates a Time of Travel sampling event.*

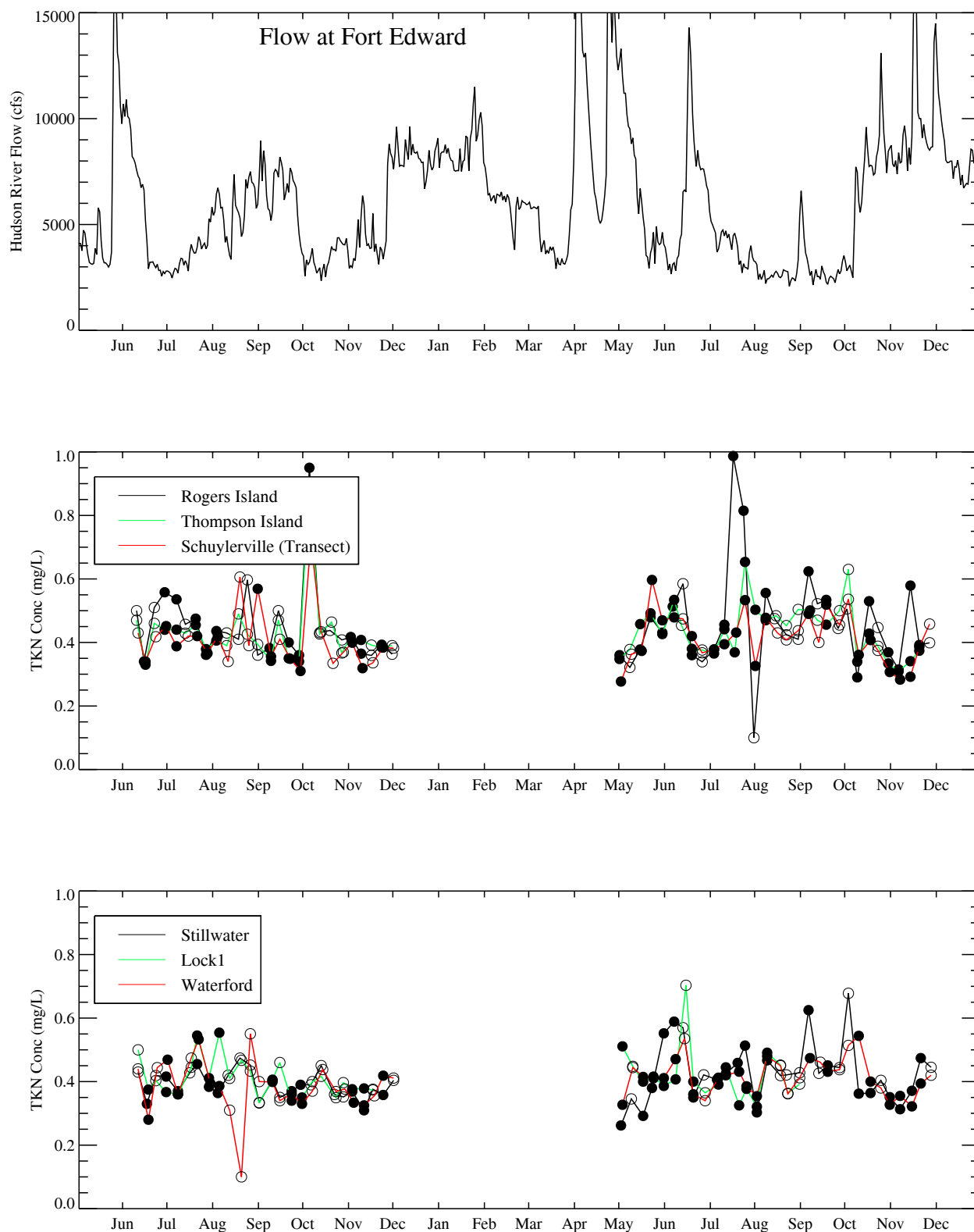
*Non-detect samples set to the MDL and plotted with open symbols.*



**Figure 1d. Spatial trends in Upper Hudson River Total Phosphorous concentrations.**

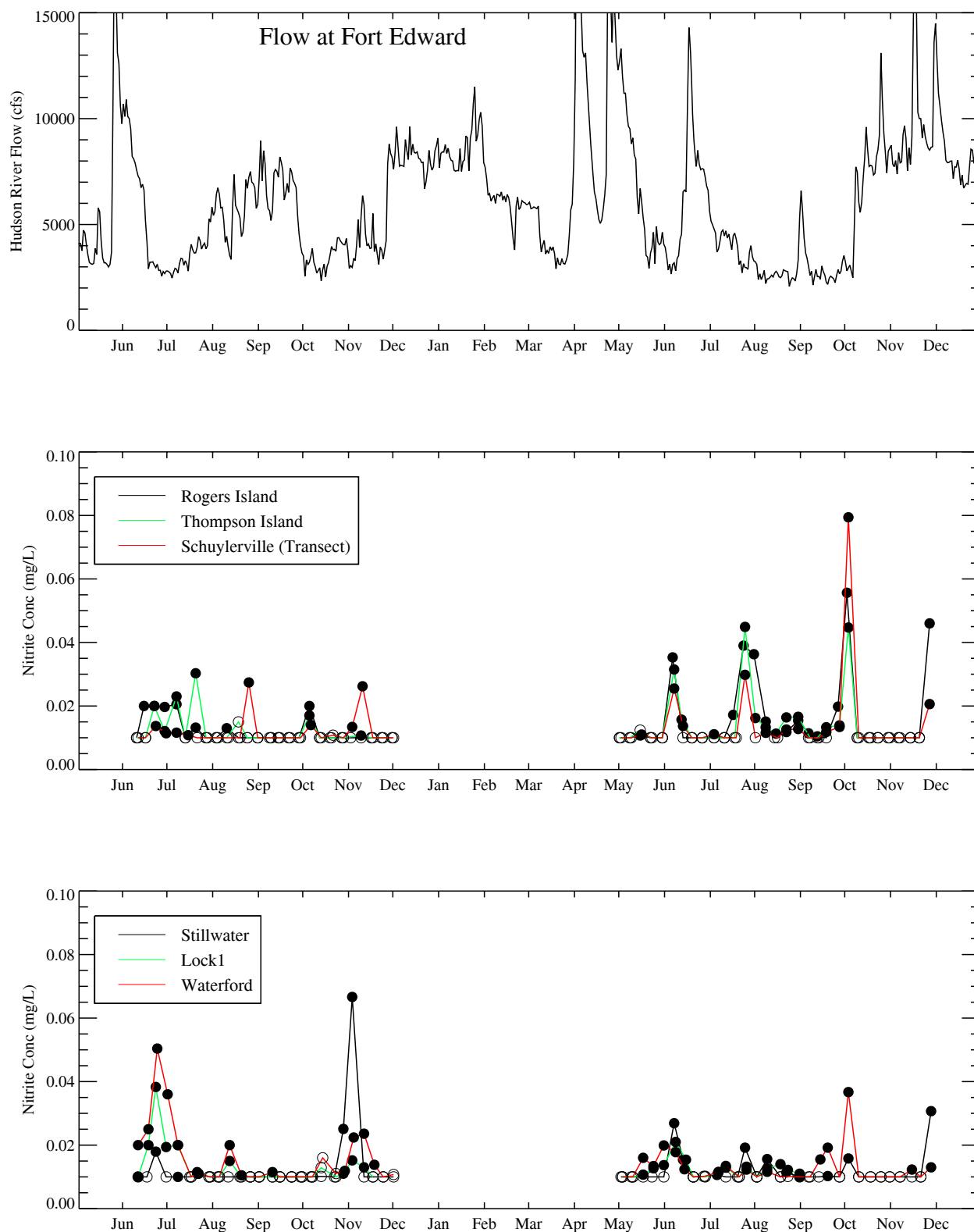
*Notes: Solid line indicates a routine sample event, dotted line indicates a Time of Travel sampling event.*

*Non-detect samples set to the MDL and plotted with open symbols.*



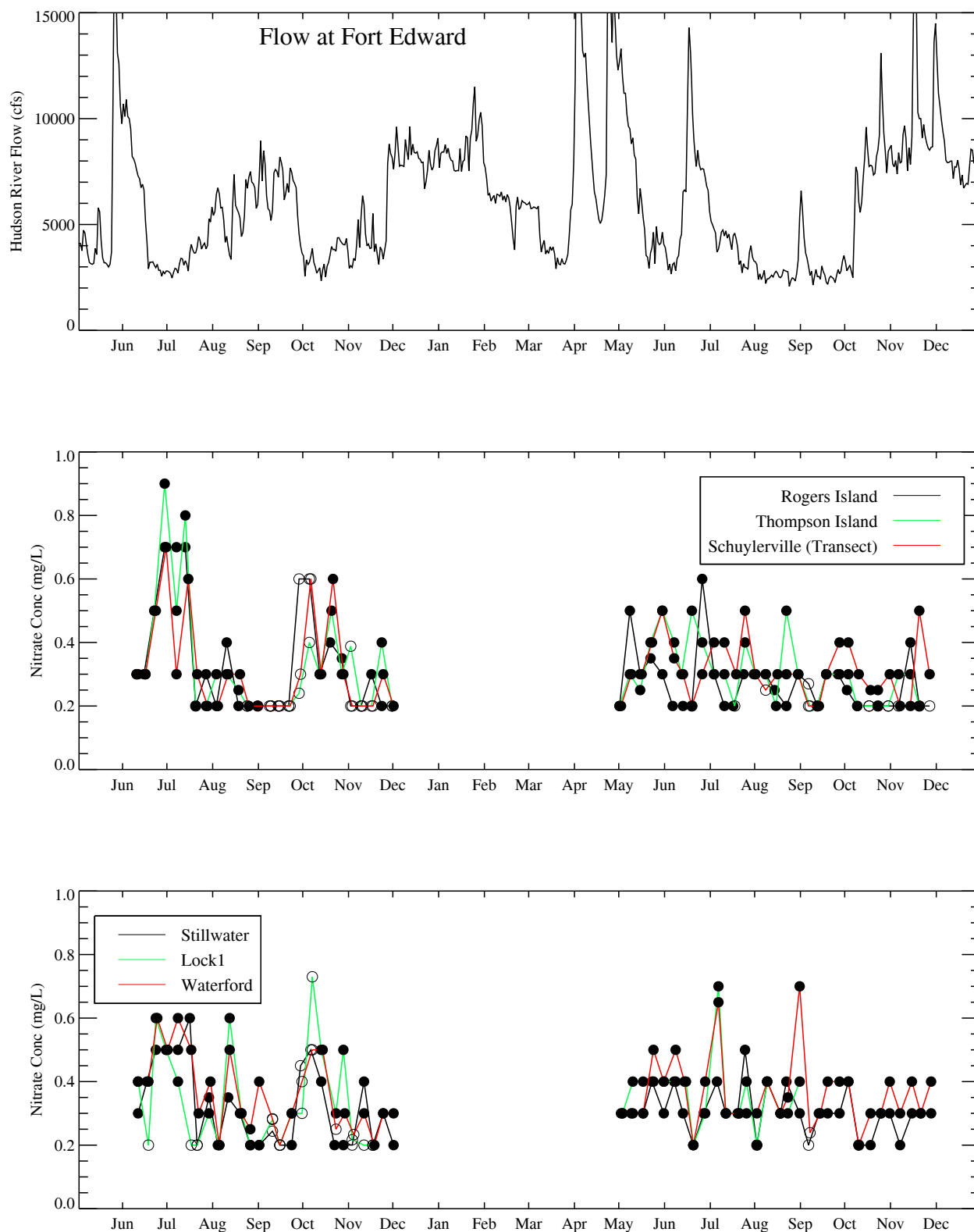
**Figure 2a. Temporal trends in Upper Hudson River TKN concentrations.**

*Notes: Flow based on USGS flow guage at Fort Edward, flow data is provisional.  
 Nutrient non-detects plotted at the detection limit with open symbols.*



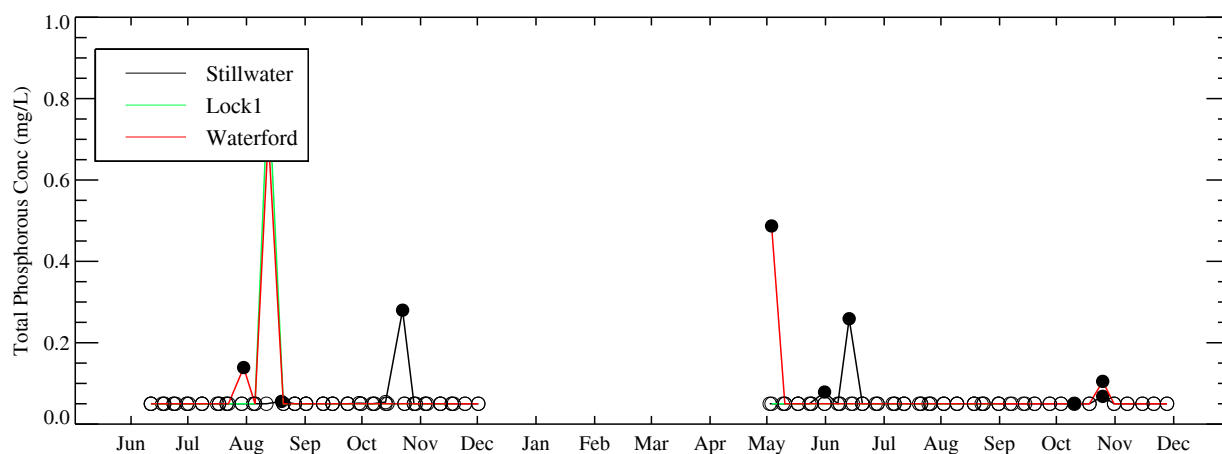
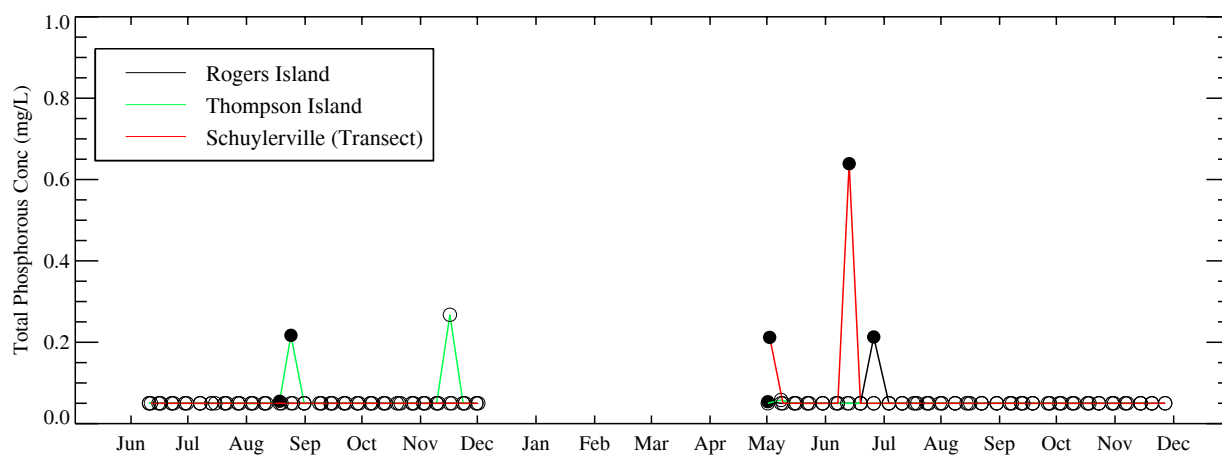
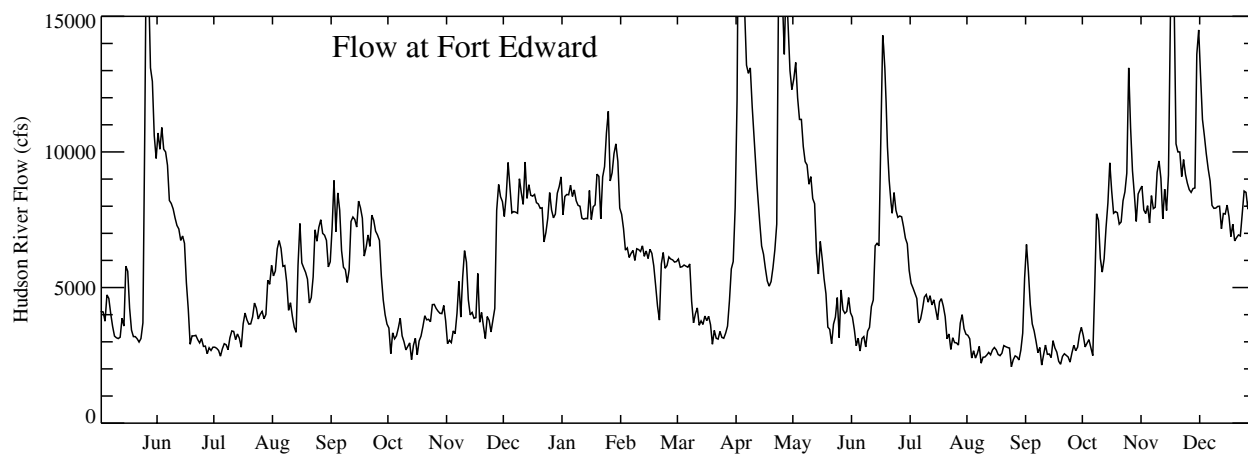
**Figure 2b. Temporal trends in Upper Hudson River Nitrite concentrations.**

*Notes: Flow based on USGS flow guage at Fort Edward, flow data is provisional.  
Nutrient non-detects plotted at the detection limit with open symbols.*



**Figure 2c. Temporal trends in Upper Hudson River Nitrate concentrations.**

*Notes: Flow based on USGS flow guage at Fort Edward, flow data is provisional.  
 Nutrient non-detects plotted at the detection limit with open symbols.*



**Figure 2d. Temporal trends in Upper Hudson River Total Phosphorous concentrations.**

*Notes: Flow based on USGS flow guage at Fort Edward, flow data is provisional.  
Nutrient non-detects plotted at the detection limit with open symbols.*

**GENERAL ELECTRIC COMPANY  
HUDSON RIVER BASELINE MONITORING PROGRAM  
CORRECTIVE ACTION MEMORANDUM NO. 6**

**Date:** May 1, 2006

**Organization Name:** Quantitative Environmental Analysis, LLC

**Initiator's Name and Title:** Christopher Yates, Project Scientist

**Problem Description:**

The BMP QAPP specifies that, samples will be collected from the historical sampling locations at Thompson Island Dam (TID-PRW2) and Schuylerville (Rt. 29 Bridge) during the first year of monitoring using techniques consistent with the historical GE sampling program (depth-integrated composite using a Kemmerer Bottle sampler). The purpose of this collection was to develop a data set of paired measurements to allow comparison of BMP data with historical data. Sampling began in June 2004 and continued through 2005, providing more data than were required by the QAPP.

Samples were collected monthly concurrent with BMP transect sampling and analyzed for PCBs. Sampling occurred year round at Schuylerville (weather permitting) and March (ice conditions permitting) through November at Thompson Island. Fourteen paired samples have been collected at Thompson Island and 16 paired samples have been collected at Schuylerville. Figure 1 presents two regressions relating data collected from historical sampling locations to the current BMP stations at Thompson Island and Schuylerville, respectively.. Both the Thompson Island paired data ( $adj-R^2 = 0.71$ ) and the Schuylerville paired data ( $adj-R^2 = 0.96$ ) demonstrate a strong correlation between the historic stations and the BMP transect locations. Figure 2 presents a probability plot of the paired differences between the historic stations and transects indicating that the differences are normally distributed and hence a Student's t-test can be used to assess the paired results. Table 1 presents the regression coefficients and statistics along with the results of the paired Student's t-tests (two-tailed). These results indicate that the calculated slope is not statistically different from one and that the intercept is not statistically different from zero at a five percent level of significance; therefore, the data collected at the historical sites are not statistically different from the data collected at the transect sampling stations.

**Reported To:** Bob Gibson, GE; John Haggard, GE; John Connolly, QEA

-----

**Corrective Action:**

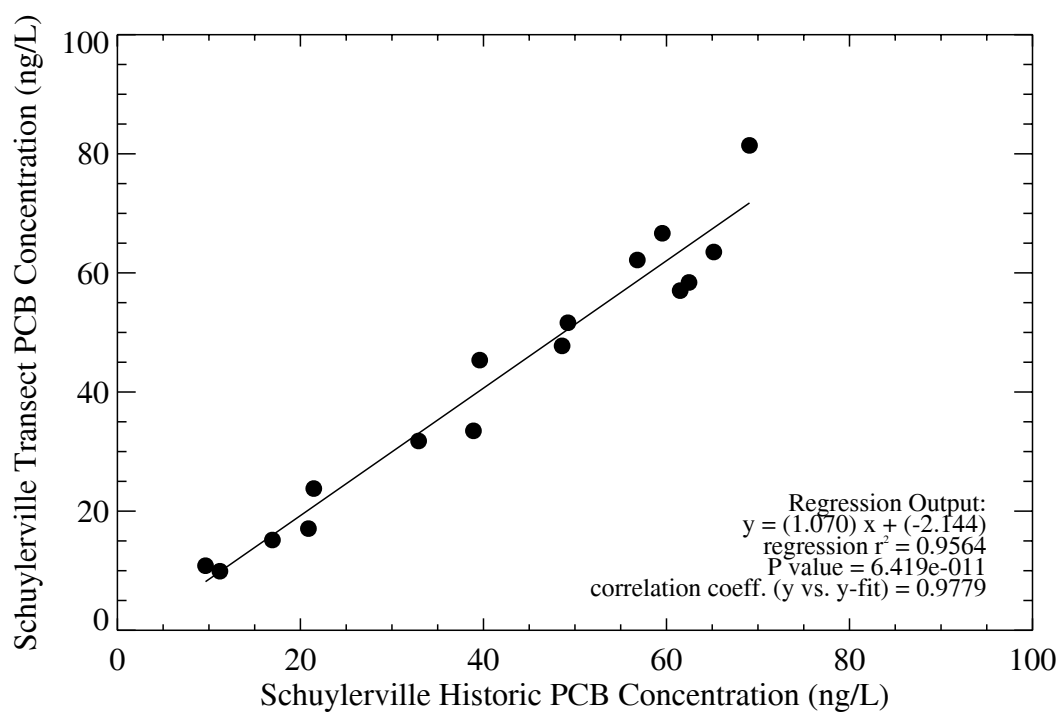
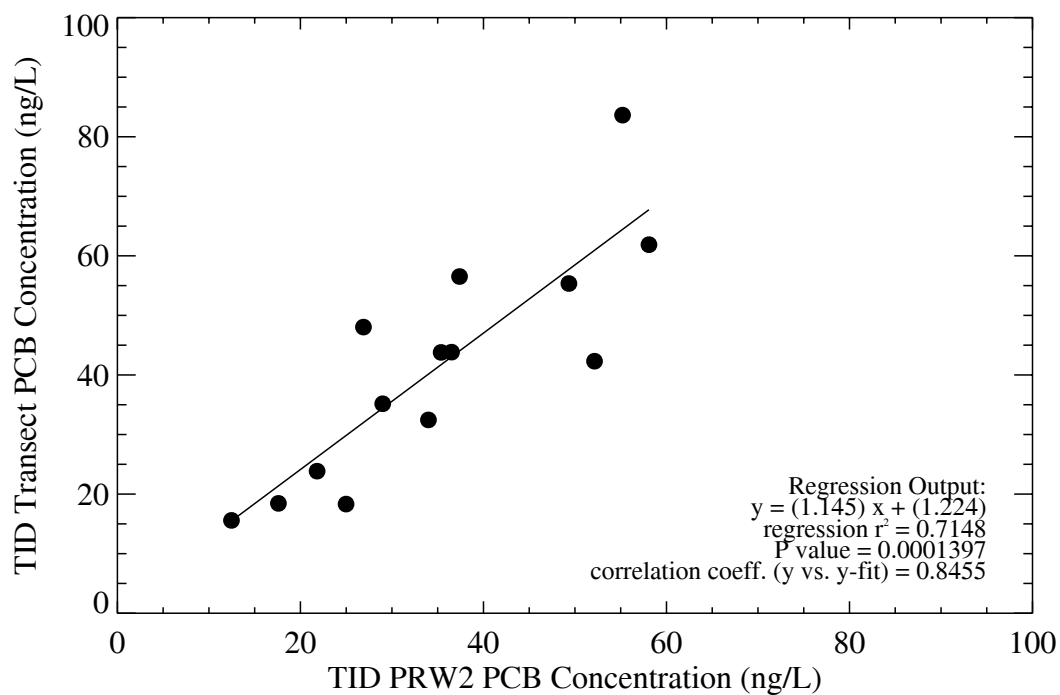
Effective May 15, 2006, GE will discontinue collecting samples at the historical locations.

**Reviewed and Implemented By:** Christopher Yates (QEA)

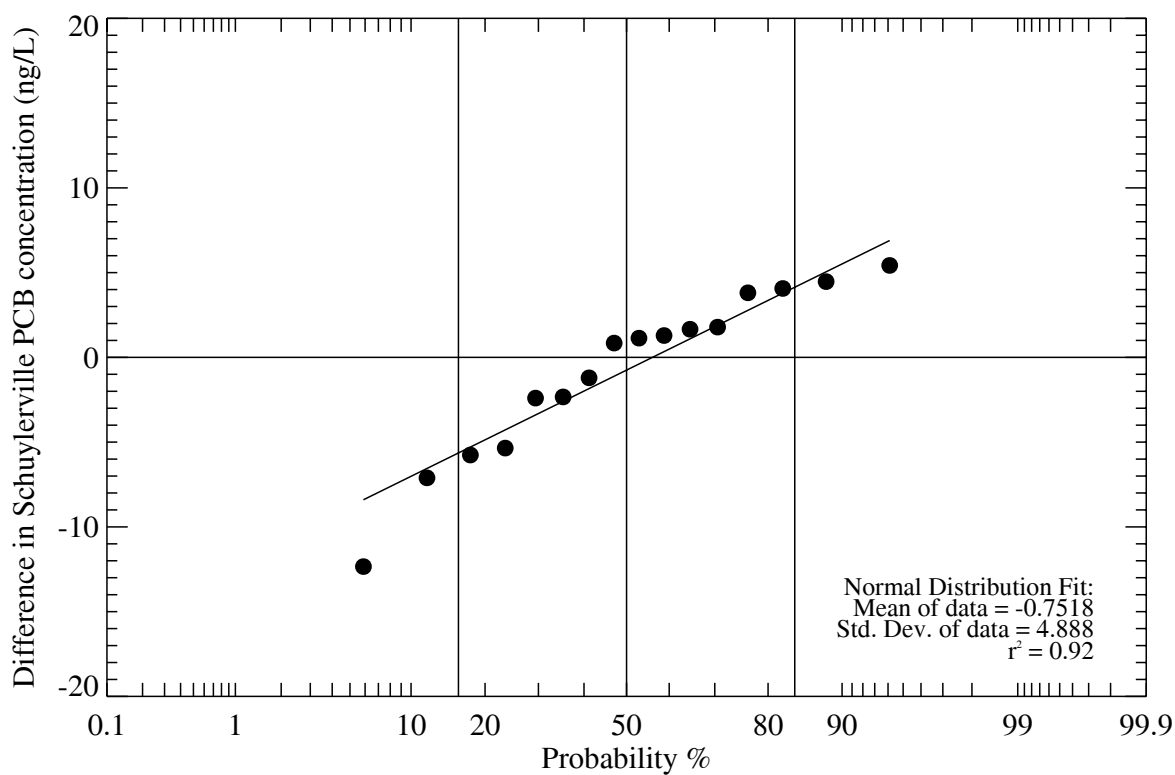
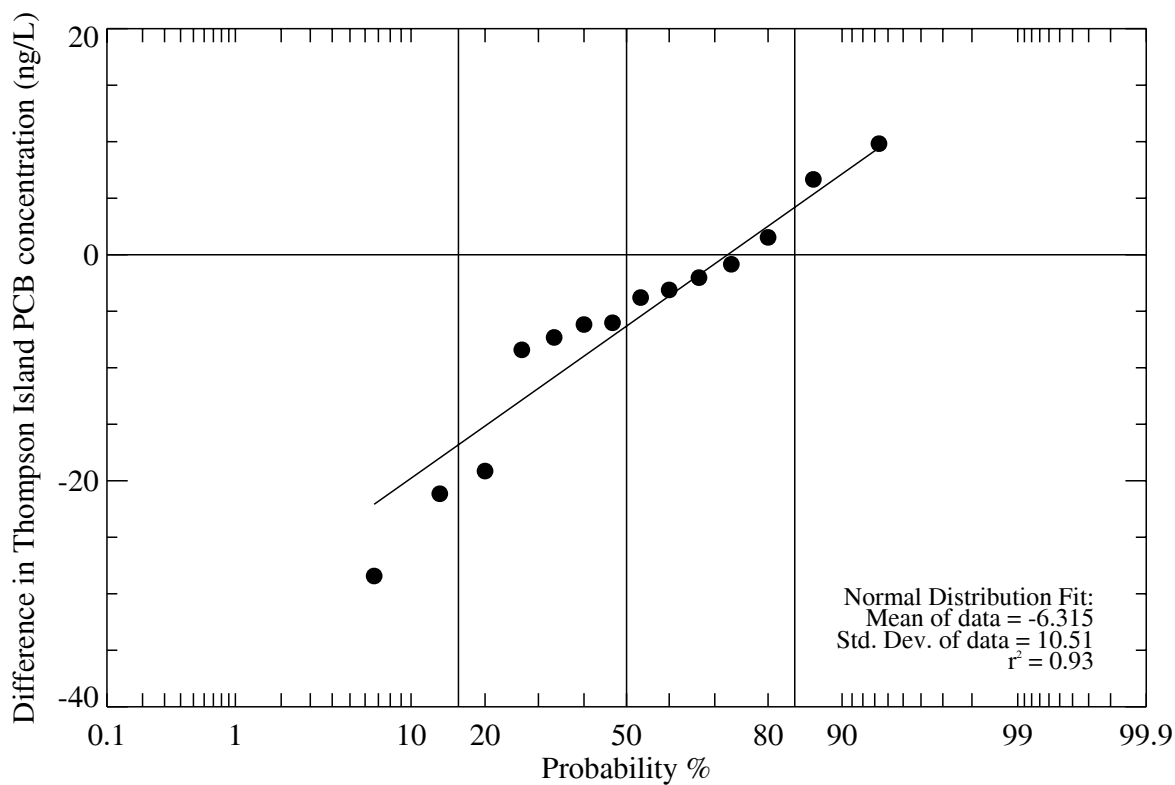
cc: GE Program Manager: John Haggard; Bob Gibson

Field Program Manager: Mark LaRue (QEA)

Other Distribution: John Connolly (QEA), Jim Rhea (QEA), Laurie Scheuing (QEA)  
David Blye (ESI); Bob Wagner (NEA)



**Figure 1. Linear regression of PCB concentration at historic stations and BMP transects.**



**Figure 2. Probability distribution of the paired difference of PCB concentrations at the historical sampling locations.**

**Table 1. Historic Station Regression Results – Paired Sampling Events 2004 and 2005.****Thompson Island - Results of 14 Paired Events (TID\_PRW2 and Transect)**

<i>Regression Statistics</i>	
Multiple R	0.845
R Square	0.715
Adjusted R Square	0.691
Standard Error	10.7
Observations	14

ANOVA					
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	3457	3457	30.0	0.00014
Residual	12	1379	114.9		
Total	13	4836			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>
Intercept	1.22	7.86	0.156	0.879
Slope	1.15	0.209	5.48	0.00014

**Intercept t-test results:** t-stat = 0.16; t-critical ( $\alpha = 0.05$ ,  $n - k = 13$ , two-tail) = 2.16.  $|t\text{-stat}| < t\text{-critical}$ , therefore we *fail* to reject  $H_0$  that the intercept = 0; the intercept is not statistically different from zero at the 5% level of significance.

**Slope t-test results:** t-stat = (test m – calculated m) / Standard Error =  $(1 - 1.15) / 0.21 = -0.71$ ; t-critical ( $\alpha = 0.05$ ,  $n - k = 13$ , two-tail) = 2.16.  $|t\text{-stat}| < t\text{-critical}$ , therefore we *fail* to reject  $H_0$  that the slope = 1; the slope is not statistically different from one at the 5% level of significance.

**Schuylerville - Results of 16 Paired Events (Center Channel and Transect)**

<i>Regression Statistics</i>	
Multiple R	0.978
R Square	0.956
Adjusted R Square	0.953
Standard Error	4.84
Observations	16

ANOVA					
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	7185	7185	306.9	6.41869E-11
Residual	14	327.7	23.4		
Total	15	7513			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>
Intercept	-2.14	2.81	-0.764	0.458
X Variable 1	1.07	0.061	17.5	6.42E-11

**Intercept t-test results:** t-stat = -0.764; t-critical ( $\alpha = 0.05$ ,  $n - k = 15$ , two-tail) = 2.13.  $t\text{-stat} < t\text{-critical}$ , therefore we *fail* to reject  $H_0$  that the intercept = 0; the intercept is not statistically different from zero at the 5% level of significance.

**Slope t-test results:** t-stat = (test m – calculated m) / Standard Error =  $(1 - 1.07) / 0.06 = -1.17$ ; t-critical ( $\alpha = 0.05$ ,  $n - k = 15$ , two-tail) = 2.13.  $|t\text{-stat}| < t\text{-critical}$ , therefore we *fail* to reject  $H_0$  that the slope = 1; the slope is not statistically different from one at the 5% level of significance.

**GENERAL ELECTRIC COMPANY  
HUDSON RIVER BASELINE MONITORING PROGRAM  
CORRECTIVE ACTION MEMORANDUM NO. 7**

**Date:** May 2, 2006

**Organization Name:** Quantitative Environmental Analysis, LLC

**Initiator's Name and Title:** Christopher Yates, Project Scientist

**Problem Description:**

Sampling is currently being performed at Bakers Falls on a weekly basis for the BMP. The BMP QAPP (p. 62) allows the sampling frequency at Bakers Falls to be reduced to monthly if the concentrations are uniformly low. Figure 1 shows a temporal plot of the PCB and TSS concentrations at Bakers Falls. The PCB concentrations are relatively consistent, with most values near or below the method detection limit of 1.1 ng/L (with the exception of two sampling events at the start of the sampling program that had higher concentrations of 4.4 and 6.8 ng/L). Some seasonal variability may exist, as detectable concentrations generally occur more frequently between May and October; however, the temporal trends are not great and reduction of the sampling frequency at this location would not compromise the ability to characterize PCB levels at this location. To illustrate this point, Figure 2 compares Tukey box plots generated using the entire data set (ignoring the outlier values at the start of the BMP program) and a subset of the data set representing a monthly sampling frequency. As can be seen, the distributions indicated by the box plots are similar. Moreover, the average PCB concentration during the May through October construction season is nearly identical for the weekly and monthly data sets, being 1.69 ng/L and 2.16, ng/L, respectively. Therefore, it is recommended to reduce the frequency of sampling at Bakers Falls once per month.

**Reported To:** Bob Gibson, GE; John Haggard, GE; John Connolly, QEA

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**Corrective Action:**

Effective June 1, 2006, GE will reduce the sampling frequency of sampling at Bakers Falls to once per month.

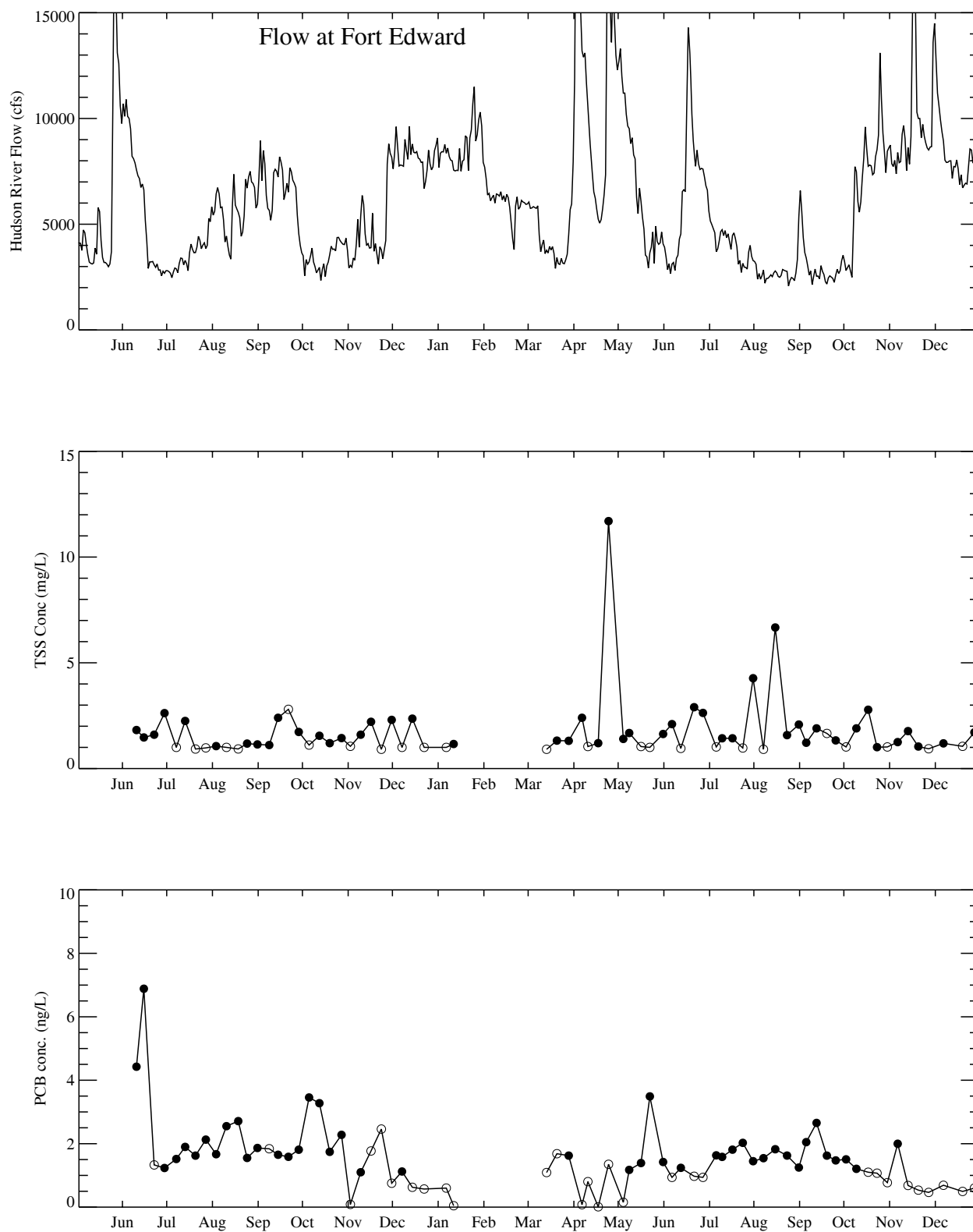
**Reviewed and Implemented By:** Christopher Yates (QEA)

cc: GE Program Manager: John Haggard; Bob Gibson

Field Program Manager: Mark LaRue (QEA)

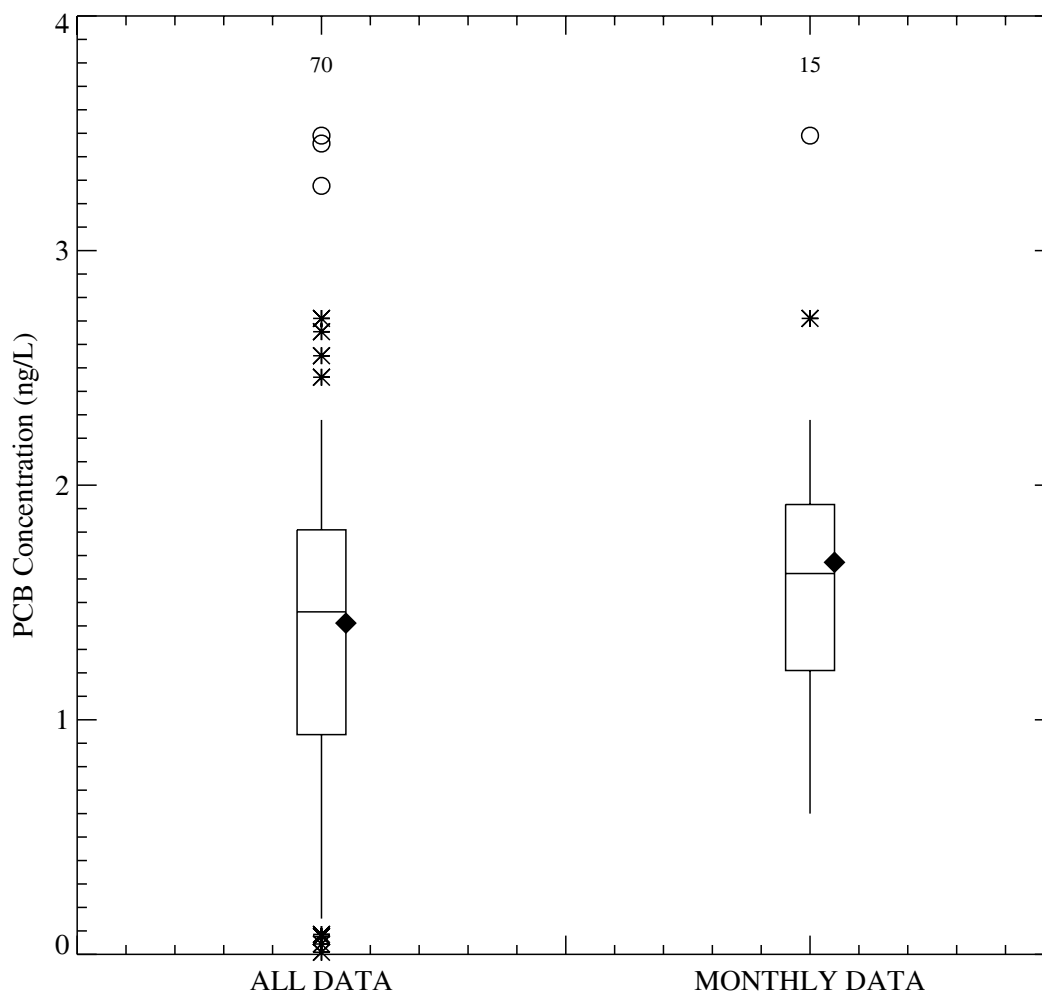
Other Distribution: John Connolly (QEA), Jim Rhea (QEA), Laurie Scheuing (QEA)

David Blye (ESI); Bob Wagner (NEA)



**Figure 1. Temporal trends in Bakers Falls PCB and TSS concentrations.**

*Notes: Flow based on USGS flow guage at Fort Edward, flow data is provisional.  
Non-detects plotted at the detection limit with open symbols.*



**Figure 2. Tukey box plot of Bakers Falls PCB data.**

*Notes:*

*The monthly data are the Bakers Falls sampling events that occurred during the routine monthly sampling weeks. Two outlier values at the start of the baseline program are excluded from this analysis.*

**GENERAL ELECTRIC COMPANY  
HUDSON RIVER BASELINE MONITORING PROGRAM (BMP)**

**Date:** May 11, 2005

**Organization Name:** Environmental Standards, Inc.

**Initiator's Name and Title:** Meg A. Michell – BMP QA Officer

**Problem Description:** STL Pittsburgh updated the laboratory's standard operating procedure (SOP) for ICP/MS metals analysis by EPA Method 200.8 that is used for the GE BMP. Most of the revisions clarify the different procedures used for the various methods covered by the laboratory's SOP (this one SOP covers ICP/MS analyses by EPA Methods 200.8, 6020, and CLP while EPA Method 200.8 is used for the GE BMP). The revisions include clarification of the procedures used for the low-level check standard ("CRQL" standard) and the internal standard recovery limits. In addition, the reporting limit (RL) for zinc was corrected and now matches the RL presented in the BMP QAPP. The changes that have been made to the SOP are minor and do not impact the ability of the method to meet the project data quality objectives (DQO's). Furthermore, STP Pittsburgh is currently performing hardness analysis for the GE BMP, which was not covered in the BMP Quality Assurance Project Plan (QAPP).

**Reported To:** Bob Gibson, GE; John Haggard, GE; John Connolly, QEA

**Corrective Action:** STL Pittsburgh's revised EPA Method 200.8 SOP and current hardness analysis SOP are attached for submission to the Agency. The attached version of the Method 200.8 SOP should replace the version included as Appendix 15 in the BMP QAPP, Revision 2, May 28, 2004. The hardness SOP is to be added to the BMP QAPP as Appendix 45.

**Approved By (USEPA RPM):** \_\_\_\_\_ **Date:** \_\_\_\_\_

**Reviewed and Implemented By:** David Blye (EnvStd).

cc: GE Program Manager: John Haggard; Bob Gibson  
QA Program Manager: David Blye (EnvStd)  
Other Distribution: John Connolly (QEA)

## **APPENDIX 15**

Control Number \_\_\_\_\_  
Implementation Date: April 15, 2005

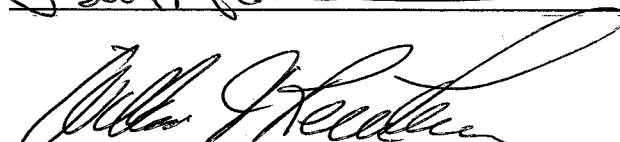
SOP No. PITT-MT-0020  
Revision No. 2.0  
Revision Date 05/19/05  
Page: 1 of 57

**STL PITTSBURGH STANDARD OPERATING PROCEDURE**  
**TITLE: ANALYSIS OF METALS BY INDUCTIVELY COUPLED PLASMA/MASS**  
**SPECTROMETRY (ICPMS) FOR METHODS 200.8, 6020 & ILM05.2**  
**(SUPERSEDES: NONE)**


Prepared by:



Reviewed by:

  
Technical Reviewer

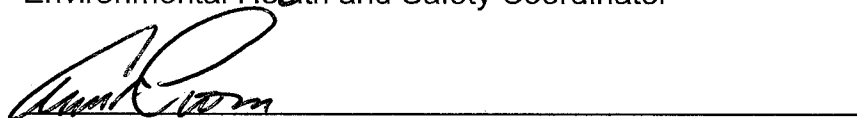
Approved by:

  
Quality Assurance Manager

Approved by:

  
Environmental Health and Safety Coordinator

Approved by:

  
Lab Director

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## 1. SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA Method 6020 and EPA Method 200.8.
- 1.2. This method is applicable to drinking, surface, and saline waters; soil and waste samples.
- 1.3. Reporting Limits  

The standard reporting limits for metals analyzed by ICP-MS are listed in Table 1. Upon client request, results below the standard reporting limit but above the current method detection limit (MDL) may be reported and qualified as "estimated".
- 1.4. Methods are based on the requirements of the US EPA Contract Laboratory Program (CLP) method ILM05.2D, and SW-846 methods 6020 and 6020. Instructions within this document that are general are given in BLACK, whilst those that apply only to 6020 are in BLUE and those that apply only to ILM05.2D are in RED.
- 1.5. Elements that may be determined using this procedure include: Al, Sb, As, Ba, Be, B, Cd, Cr, Co, Cu, Fe, Pb, Mn, Mo, Ni, Se, Si, Ag, Sr, Tl, Sn, Ti, V, Zn, Ca, Mg, K, and Na.  

Note: successful Ag analysis may require all solutions to be prepared as described, but with the addition of hydrochloric acid to 1% (v/v). This may degrade performance for As, Se and V.

## 2. SUMMARY OF METHOD

- 2.1. The sample solution is introduced into a pneumatic nebulizer via a peristaltic pump. The nebulizer generates a fine aerosol by bringing the solution into contact with a high velocity flow of argon gas at its tip. The nebulized sample is sorted by droplet size in the spray chamber. Large droplets are rejected, whilst smaller particles are transported with the gas stream into the plasma.
- 2.2. The argon plasma operates with a continuously applied radio frequency (RF) field to give a high-energy discharge consisting of argon atoms, ions and electrons. The hottest part of the plasma can attain 6000-8000 K. In the plasma, aerosol droplets undergo evaporation, atomization and ionization. Ions are sampled through an aperture in a metal cone (sampler) at atmospheric pressure, into the expansion region at about 2 mbar and subsequently through an aperture in a second metal cone (skimmer) into the intermediate chamber.
- 2.3. An electrostatic ion lens system focuses the ion beam through a differential aperture into the analyser chamber, at about 10<sup>-7</sup> mbar. The ions are filtered by mass-to-charge ratio in microsecond timescales by the quadrupole. The selected mass is detected by a discrete dynode electron multiplier. The multiplier has two simultaneous modes of operation: pulse count and analogue. The combination of these two modes allows seamless detection spanning 8 - 9 orders of magnitude. A detector "cross-calibration" is required for the analogue counts to be converted to equivalent pulse counts. The output from the detector is proportional to the concentration of the element in the aspirated solution, hence the concentration of unknown samples may be calculated when the instrument response is calibrated with standards of known concentration.
- 2.4. The linear range may vary from instrument to instrument and is dependant upon the sensitivity determined by the optimization parameters. This should be determined by the individual laboratory. In the test study at STL Pittsburgh, the linear ranges listed in Table 1 were obtained:

2.4.1. Table 1. Test study linear ranges for the X5 ICP-MS

Analytes	Linear Range (mg/L)
Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, V, Zn	0.20 – 20.0
Al, Ca, Mg, K Na, Fe	100 - 1500

2.5. Calibration standard concentrations are listed in Table 2.

2.5.1. Table 2. Calibration standard concentrations for analysis of water and waste

Analytes	Calibration Range (mg/L)
Al,	1.0
Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, V, Zn	0.20
Ca, Mg, K Na, Fe	100
Fe	50
B, Mo, Sn, Sr, Ti	0.20
Si	10

### 3. DEFINITIONS

3.1. See the LQM for definitions of general terms

3.2. See appendix for Glossary of Abbreviations

### 4. INTERFERENCES

4.1. Isobaric Isobaric interferences. Elemental isobaric interferences occur when different elements have isotopes at the same nominal mass, e.g.  $^{114}\text{Cd}$  and  $^{114}\text{Sn}$ . Problematic elemental isobaric interferences for these methods are listed in Table 3. The correction factors given in Table 3 are based on theoretical isotopic abundance ratios and may require adjustment.

Table 3 Isobaric Interferences and Correction Equations

m/z	Analyte	Interferent	Correction
58	Ni	Fe	$58\text{Ni}=58\text{M}-0.0040*56\text{Fe}$
64	Zn	Ni	$64\text{Zn}=64\text{M}-0.0440*60\text{Ni}$
82	Se	Kr	$82\text{Se}=82\text{M}-1.0010*83\text{Kr}$
114	Cd	Sn	$114\text{Cd}=114\text{M}-0.0270*118\text{Sn}$
115	In	Sn	$115\text{In}=115\text{M}-0.0140*118\text{Sn}$

123	Sb	Te	$123\text{Sb} = 123\text{M} - 0.1240 \cdot 125\text{Te}$
138	Ba	Ce	$138\text{Ba} = 138\text{M} - 0.0030 \cdot 140\text{Ce}$

- 4.2. Abundance Sensitivity - Abundance sensitivity is the ability of the quadrupole to separate a low intensity peak from an adjacent high intensity peak. An example of the requirement of this is the detection of low concentrations of manganese ( $m/z$  55) in the presence of high concentrations of iron ( $m/z$  56). Quadrupole resolution and bias can be adjusted during set-up to resolve these signals.
- 4.3. Isobaric Polyatomic Ion Interferences - Polyatomic ions are produced by chemical reaction in the plasma and the interface region. If these polyatomic ions have the same nominal mass to charge ( $m/z$ ) ratio as an analyte a polyatomic interference is observed. The principle polyatomic species for this method are listed in Table 4. Some of the correction factors given in Table 4 are based on theoretical isotopic abundance ratios and may require adjustment. Other factors were derived empirically. The stability of the empirical factors was determined during the test study at Thermo Electron. It was found that the factors require little or no adjustment and can be transferred between similarly configured X5 instruments.

**Table 4. Isobaric Polyatomic Interferences and Correction Equations**

$m/z$	Analyte	Interferent	Correction
51	V	ClO	$51\text{V} = 51\text{M} - 3.0460 \cdot 53\text{ClO}$ $53\text{ClO} = 53\text{M} - 0.114 \cdot 52\text{Cr}$
52	Cr	ArC, ClOH	$52\text{Cr} = 52\text{M} - 0.0050 \cdot 13\text{C}$
56	Fe	CaO	$56\text{Fe} = 56\text{M} - 0.1500 \cdot 43\text{Ca}$
56	Co	CaO, CaOH	$59\text{Co} = 59\text{M} - 0.0046 \cdot 43\text{Ca}$
60	Ni	CaO	$60\text{Ni} = 60\text{M} - 0.0020 \cdot 43\text{Ca}$
75	As	ArCl	$75\text{As} = 75\text{M} - 3.000 \cdot 77\text{ArCl}$ $77\text{ArCl} = 77\text{M} - 0.8000 \cdot 82\text{Se}$ $82\text{Se} = 82\text{M} - 1.0010 \cdot 83\text{Kr}$
111	Cd	MoO	$111\text{Cd} = 111\text{M} - 0.9820 \cdot 108\text{MoO}$ $108\text{MoO} = 108\text{M} - 0.712 \cdot 106\text{Cd}$

- 4.4. Physical Interferences - Physical interferences include transport effects, ionization effects and deposition effects in the sample introduction system, plasma and interface, which result in signal suppression and signal drift. Transport effects arise from variations in solution properties, e.g. viscosity or surface tension, which affect nebulization efficiency and aerosol droplet size. The concentration of dissolved matter will affect the ionization efficiency of the analytes in the plasma and will cause a mass-dependant suppression effect and contribute to space-charge effects. Dissolved matter may also condense on the cones, altering the ion beam profile. This normally manifests itself as a time-dependant downward signal drift. To reduce the severity of these effects it is advised that the total dissolved solids concentration

of solutions aspirated should be limited to <0.05%. Samples known to contain higher dissolved solids concentrations should be diluted. Signal suppression and drift can be corrected, to a degree, with the use of internal standardization techniques. Since these effects can be mass-dependant and may be related to the ionization potential of the element, a multiple-element internal standard approach should be used.

- 4.5. Memory Effects - Memory effects occur when the signal for an analyte from a sample contributes to the signal of a subsequent sample. This effect can be severe for certain elements due to their physico-chemical properties, e.g. mercury. This effect is minimised by aspirating a wash solution between samples. A monitored wash can be used in order to ensure that analyte signals recover to the background level.

## 5. SAFETY

- 5.1. Procedures shall be carried out in a manner that protects the health and safety of all associates.
- 5.2. Eye protection that satisfied ANSI Z87.1 (as per the Corporate Safety Manual), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.
- 5.2.1. Neoprene, NDex (nitrile), and TRIonic® Cleanroom gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.
- 5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.
- 5.3.1. The following materials are known to be corrosive: sulfuric acid; hydrochloric acid; and nitric acid.
- 5.3.2. The following materials are known to be oxidizing agents: nitric acid; hydrogen peroxide.
- 5.3.3. The waste pumped from the spray chamber is corrosive and must be handled with care, especially if large volume containers are used, as these may be heavy and awkward to carry. Empty the waste vessel daily to reduce the quantity that must be disposed each time and to keep weight to a minimum. Protective clothing, including hand and eye protection must be worn when handling this waste.
- 5.3.4. The wash solution is corrosive and must be handled with care. This solution must be prepared and stored in a vessel made of a robust acid-resistant material with a tight fitting lid that it is resistant to breakage if dropped. Large volumes of this solution will be heavy and may be awkward to carry. Ensure adequate provision for transporting the vessel, i.e. suitable handles on the vessel, minimum distance between the preparation area and the instrument. Use a cart to transport the vessel where necessary or ask for assistance in carrying.
- 5.3.5. Many of the concentrated metal standard solutions are toxic and must be handled with care. Skin and eye protection should be worn when handling and inhalation of vapours must be prevented.
- 5.3.6. Fumes generated by the plasma can be hazardous and must be removed from the laboratory with an extraction system as detailed in the X Series site planning

guide. If the extraction system is faulty do not attempt to use the instrument. The extraction system should be inspected on a regular basis.

- 5.3.7. The plasma emits strong UV light and is harmful to vision.
- 5.3.8. **WARNING:** AVOID looking directly at the plasma.
- 5.3.9. The plasma is a source of radio frequency (RF) radiation and intense, ultra-violet radiation that can damage the eyes. This radiation is normally contained by the system, but operators must be aware of the dangers. The instrument must be properly maintained by qualified service personnel. Never attempt to defeat hardware interlocks – they are there for your safety.
- 5.3.10. **WARNING:** People with pacemakers should not go near the instrument while in operation. DIAZOMETHANE is an extremely toxic gas with an explosion potential. Since the explosion potential is catalyzed by imperfections in glass, generation of diazomethane must be carried out in glassware free from etches, cracks, chips, and which does not have ground glass joints. Solutions of diazomethane will be kept at temperatures below 90°C. Diazomethane must be generated and handled in a fume hood.  
Note: Diazomethane has not been classified as a carcinogen under the current OSHA definition.
- 5.3.11. Should the plasma need to be extinguished in an emergency, open the torch box door. This will immediately cut-off the power to the plasma RF generator, extinguishing the plasma. After extinguishing the plasma, the torch, torch box, cones and cone housing may remain very hot for some time. Operators must be aware of this fact and allow cooling time prior to handling these components.
- 5.3.12. There are high voltage components inside the instrument. Routine maintenance does not require access to any of the electronic components. If an electronic fault is suspected, a qualified service engineer must be called. Do not attempt to tamper with electronic components yourself.
- 5.4. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.
- 5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported immediately to a laboratory supervisor.

## 6. EQUIPMENT AND SUPPLIES

- 6.1. X Series ICP-MS fitted with Xi interface and Y-connector for on-line internal standard addition (supplied with this package).
- 6.2. Cetac ASX-510 autosampler.
- 6.3. Ultrapure water system capable of delivering de-ionized, polished water of at least 18 MΩ cm
- 6.4. Yellow/orange tab peristaltic pump tubes (~0.5 mm ID)

- 6.5. White/white tab peristaltic pump tubes (~1 mm ID)
- 6.6. A range of adjustable pipettes, such as Rainin pipettes. Adjustable pipettes with a capacity of 0.1 mL, 1 mL, and 10 mL are recommended. These must be calibrated regularly to ensure accurate volumes are delivered.

## 7. REAGENTS AND STANDARDS

### 7.1. General Reagents

- 7.1.1. **Laboratory Water** - All laboratory water used in these procedures must be of very high quality, purified with a reverse osmosis system and polished with an ion exchange system to give a final product of resistivity >18 MΩ cm.
- 7.1.2. **Hydrochloric Acid** (sp. gr. 1.18) - Hydrochloric acid must be at least Romil "SPA", J.T. Baker "Instra Analyzed", BDH/Merk "Analar", Fisher "Optima" - grade or equivalent. Hazards – corrosive, causes severe burns.
- 7.1.3. **Nitric Acid** (sp. gr. 1.42) - Nitric acid must be at least Romil "SPA", J.T. Baker "Instra Analyzed", BDH/Merk "Analar", Fisher "Optima" - grade or equivalent. Hazards – oxidising and corrosive, causes severe burns.
- 7.1.4. **2 % (m/v) Nitric Acid** - This reagent is used for the calibration blank, ICB, CCB, sample dilution and solution preparation. Add 5 mL of Conc of HNO<sub>3</sub> to DI water and dilute to 250 mL

## 8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. Samples are to be collected in plastic or glass containers.
- 8.2. All soils must be refrigerated to 4°C ± 2°C.
- 8.3. The analytical holding time for metals by ICP-MS is 6 months.
- 8.4. Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure. Method 200.8 has its own digestion specifications which are followed by the laboratory. Method 3005A is used for total recoverable metals and method 3010A is used for total metals by 6020. These are covered in the SOP C-IP-003. Upon consultation with the client dissolved samples can forego digestion to help prevent contamination when very low detection limits are required.
- 8.5. Soil or waste samples should be digested before analysis using an appropriate digestion procedure. Method 3050B of SW846 is the appropriate digestion procedure. The SOP for 3050B is C-IP-0002.

## 9. QUALITY CONTROL

- 9.1. Initial Demonstration of Capability
  - 9.1.1. For the standard analyte list, the initial demonstration IDC and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin.
  - 9.1.2. For new analytes an MDL study should be performed and calibration curve generated before analyzing any samples.
- 9.2. Control Limits

- 9.2.1. Control limits are utilized for matrix spikes and laboratory control samples (LCS). These limits must be reviewed at least annually against current data.

QC Type	200.8	6020	ILM05.2
LCS	85 – 115	80 – 120	80 – 120
MS	70 – 130	75 – 125	75 – 125
RPD	± 20	± 20	± 20

- 9.2.2. All LCS and MS recoveries must be entered into QuantiMS or other database so that accurate historical control charts can be generated. For tests without a separate extraction, matrix spikes will be reported for all dilutions.
- 9.2.3. Refer to the QC program document (QA-003) for further details regarding control limits.

### 9.3. Quality Control Batch

The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike/matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify particular samples for MS/MSD, the batch may contain multiple MS/MSDs. See policy QA-003 for further definition of the batch.

### 9.4. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if precision data is required by the client. The LCS pair is then evaluated according to the MS/MSD RPD criteria. Use of a LCS pair in place of a MS/MSD must be documented using Clouseau.

### 9.5. Method Blank

One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher. Certain programs, such as USACE, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit.

- If the analyte is a common laboratory contaminant (copper, iron, zinc), the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than five times the RL. Such action must be documented in the NCM program.
- Re-preparation and reanalysis of any samples with reportable concentrations of analytes less than 10 times the value found in the method blank is required unless other actions are agreed with the client.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported. This must be documented in the NCM program.
- If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all positive results in associated samples are flagged with a "J", and appropriate comments may be made in a narrative to provide further documentation.

- 
- 9.5.1. Refer to the QC Program document (QA-003) for further details of the corrective actions.
- 9.5.2. For dissolved metals samples which have not been digested or matrix matched; a CCB result is reported as the method blank. The CCB analyzed immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.
- 
- 9.5.3. Methodologies for MDL assessment are detailed in SW-846 Chapter 1, method 6020 and in 40 CFR Part 136 Appendix B.

9.6. Laboratory Control Sample (LCS)

- 9.6.1. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless otherwise requested by the client.
- 9.6.2. If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur:
- Check calculations,
  - Check instrument performance,
  - Reanalyze the LCS, and if still outside of control limits,
  - Evaluate the data, and/or
  - Re-prepare and reanalyze all samples in the QC batch.
- 9.6.3. Data may be reported with an anomaly in the following cases:
- The LCS recoveries are high and the analyte of concern is not detected in field samples,
  - All target requested analytes are within control, but other LCS compounds are out of control,
  - If no sample preparation is performed (eg, dissolved metals), the LCS may be reprepared and reanalyzed within the same sequence.
- 9.6.4. The analyst should evaluate the anomalous analyte recovery for possible trends.
- 9.6.5. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.
- 9.6.6. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6.7. For dissolved metals samples which have not been digested or matrix matched, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same analytes as the LCS (See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the historically generated limits.

Note: Some programs require an Matrix Spike and Matrix Replicate in lieu of an MS/MSD. When a matrix spike/matrix replicate is performed the matrix spike is evaluated for accuracy (% recovery) and the matrix replicate is evaluated for precision (RPD).

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the matrix spike/spike duplicate and the LCS, the process is out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If a MS/MSD or MS/Dup is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7.1. If the amount of an analyte found in the unspiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply and recoveries are not evaluated. Other analytes in the MS and MSD must still be reported. File an NCM stating that the 4X rule was applied, and report the recovery in the LIMS as "ND MSB". This NCM must be included in the final report.

9.7.2. For dissolved metals samples which have not been digested or matrix matched, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample.

9.8. Linear Range Verification (LR) - The linear range is determined semi-annually (2x/year) for each element on the standard list. See section 13 for details of the linear range verification. The Linear Range study must be performed quarterly if doing ILM05.2.

9.9. The internal standard intensities in samples must be within 60 to 125% of the IS intensities for the Calibration Blank for method 200.8 and from 30% to 120% for method 6020. If this criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

9.9.1. For method 6020 the internal standard intensity in the ICB, CCB and CCB should be within 20% of the IS intensity in the calibration blank of the initial calibration. If not, the analyst should check for any instrument anomalies and continue if none are noted. For method 200.8 the IS acceptance range does not

vary from the 60 to 125% noted above.

- 9.10. Interference Check Solutions (ICSAs) - The results of ICSA must be within  $\pm 3\text{CRQL}$  of the analytes "true" value or  $\pm 20\%$  of the analytes "true" value, whichever is the greater. The "true" value will be taken as zero, unless otherwise indicated in the solution manufacturer's literature. The software automatically checks for compliance with the above, based on a "true" value of zero. If a result falls outside this range, the analysis must be terminated and the samples associated must be reanalyzed.
- 9.11. Interference Check Solution Spike Recoveries (ICSABs) - Results of ICSAB must be within  $\pm 20\%$  of the analytes "true" value. The software automatically checks for compliance with the above, based on the values indicated in (6.5.2 or 6.5.4). If a result falls outside this range, the analysis must be terminated and the samples associated must be reanalyzed.
- 9.12. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV must fall within  $\pm 10\%$  of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within  $\pm$  the reporting limit (RL) from zero. (Certain programs, may require a more stringent evaluation of ICB, for instance, that the blank not contain any analytes of interest at a concentration greater than  $\frac{1}{2}$  the reporting limit.) If either the ICV or ICB fail to meet criteria, the analytical sequence should be terminated, the problem corrected, the instrument recalibrated and the calibration re-verified.
- 9.13. CRQL Check Standard (CRI)  
**FOR ILM05.2, THE RESULTS OF THE CRI MUST BE WITHIN THE RANGE 70-130% RECOVERY FOR ALL ANALYTES, EXCEPT CO, MN AND ZN, WHICH MUST BE IN THE RANGE 50-150% RECOVERY. THIS IS CHECKED BY THE SOFTWARE, BASED ON THE VALUES GIVEN IN (6.6.3). IF ANY ANALYTE IS OUTSIDE THE RANGE INDICATED, THE SAMPLE MAY BE RE-RUN ONCE. IF THE RESULTS FALL WITHIN THE REQUIRED VALUES UPON RE-RUN, NO FURTHER CORRECTIVE ACTION NEED BE TAKEN. IF STILL OUTSIDE THE ACCEPTABLE RANGE, THE ANALYSIS SHALL BE TERMINATED, THE PROBLEM CORRECTED AND THE SAMPLES REANALYZED. FOR NON CLP METHODS THE METHOD DOES NOT SPECIFY CRITERIA, HOWEVER THE LAB USES THE RANGE 50 – 150%.**
- 9.14. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. Results for the CCV must be within the range 90-110% recovery. This is checked by the software, based on the values in (6.6.2). If outside this range, the analysis must be terminated, the problem corrected and the samples since the last valid CCV must be re-analyzed.. The CCB result must fall within  $\pm$  RL from zero. (Certain programs, may require a more stringent evaluation of the CCB, for instance, that the blank not contain any analytes of interest at a concentration greater than  $\frac{1}{2}$  the reporting limit. The analyst should refer to the project notes provided by the PM to identify when this is an issue and if so what the corrective actions to take for exceedances) Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, the CCV or CCB may be reanalyzed once and accepted if there is a reason for the initial out-of-control event such as carryover from a high concentration sample. Otherwise, if the CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.9 for an illustration of the appropriate rerun sequence).
- 9.15. Post-Digestion Spike Samples (PDS) A post digestion spike will be run on a sample if the

MS/MSD for the sample falls outside of % recovery criteria. A post digestion spike is a matrix spike on a sample, which is added after the sample preparation is completed. For 6020 the default matrix spike protocol is a "post digestion spike". However, STL Pittsburgh will perform a conventional matrix spike and spike duplicated as the default matrix QC. We will perform the "PDS" only where the conventional matrix spike fails. We believe that this approach will provide more complete matrix information than the default requirements. The spike recovery from the post digestion spiked sample should be within the range 75-125% where the spike value is greater than 25% of the indigenous analyte concentration. The software calculates this based on the following equation:

$$\% \text{Repeatability} = 100 * (\text{Spk-Orig}) / \text{Tru}$$

where, Spk is the spiked sample result and Orig is the original sample result and Tru is the True spiked concentration value. If a result is outside the required range, the data should be assessed carefully and samples may require reanalysis.

- 9.16. Serial Dilution Samples (SER) - Some regulatory programs such as require a dilution test be performed for each matrix within an analytical batch determination. The results of the serial dilution sample(s) (SER) after dilution correction should be within the range 90-110% of the original sample, if the result for the original sample is greater than 50\*IDL for CLP or greater than 50\*MDL for 200.8 or 6020.

- 9.17. The software calculates this based on the following equation:

9.18.  $\% \text{Repeatability} = 100 * \text{Ser} / \text{Orig}$

- 9.19. where, Ser is the dilution corrected serial diluted sample result and Orig is the original sample result. If a result is outside the required range, the data should be assessed carefully and samples may require reanalysis.

- 9.20. Duplicate Samples (DUP); %RPD =  $\pm 20\%$  : Results of the duplicate sample(s) (DUP) must be within  $\pm 20\%$  of the results of the original sample, where the result is greater than or equal to 5\*CRQL for CLP or greater than 5\*RL for 200.8 or 6020. The software calculates this based on the following equation:

$$\% \text{RPD} = (S - D) / [(S + D) / 2] * 100\%$$

where, D is the duplicate sample result and S is the original sample result.

If a result is outside the required range, the data should be assessed carefully and samples affected may need to be reanalyzed where the project requires it.

- 9.21. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the QA Manager.

- 9.22. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

- 9.23. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

## 10. CALIBRATION AND STANDARDIZATION

### 10.1. Instrument start-up

- 10.1.1. Follow the instrument start-up procedure outlined in the Thermo X-Series ICP-MS Operator's Manual.

### 10.2. Instrument Tuning

- 10.2.1. Aspirate a 20 ppb tuning solution containing all of the tuning elements. The 6020 tuning elements are Li, Co, In, and Tl. The instrument manufacturer monitors Mg, Ce, Be & Pb for instrument performance.
- 10.2.2. Mass calibration and resolution checks must be documented and included as part of the raw data package.
- 10.2.3. Resolution must be  $< 0.90$  amu at 10% peak height for the 6 tuning (Be, Ce, Co, In, Mg, & Pb) for 6020. Resolution must be  $\leq 0.75$  amu at 5% of the peak height for EPA 200.8 and ILM05.2. And the resolution must be  $\leq 0.9$  amu at 5% of the peak height for Method 200.8.
- 10.2.4. Mass calibration must be within  $\pm 0.1$  amu from the actual value for the 6 tuning elements (Be, Ce, Co, In, Mg, & Pb) or the mass calibration must be adjusted.
- 10.2.5. A "daily" performance check must be performed. This uses the same tuning solution as above. The 6 tuning elements must have RSDs below 5%. The oxides must be below 3.5%. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

### 10.3. Initial Calibration

- 10.3.1. Calibration consists of a blank and the following calibration standards (STD, STD 2X, and STD 3X see Table 2 for concentrations) in accordance with the manufacturer's procedure. Use the average of three integrations for both calibration and sample analyses.
- 10.3.2. Following the STD, STD2X & STD3X, an ICV/ICB pair is analyzed. The ICV must be within  $\pm 10\%$  of the true value to be acceptable.
- 10.3.3. For 6020 and ILM05.2, following the ICV/ICB pair, the CRI/RLV is run then the ICSA is analyzed.
- 10.3.4. For 6020 and ILM05.2, following the ICSA, analyze the ICSAB. The ICSAB must be within  $\pm 20\%$  of the true value.
- 10.3.5. Internal standards are added to all standards and samples by the instrument automatically prior to analysis.

### 10.4. Continuing Calibration:

- 10.4.1. Following every 10 samples (including lab QC), analyze a CCV/CCB pair. These must be within  $\pm 10\%$  of the true value for analysis to continue. For methods 6020 and ILM05.2, a CCV/CCB pair should also be analyzed immediately after the ICSAB.
- 10.4.2. All samples must be bracketed by an acceptable CCV/CCB pair. Where a CCV/CCB fails the samples preceding it back to the last acceptable CCV/CCB must be reanalyzed.

## 11. PROCEDURE

### 11.1. Instrument Set-up

- 11.1.1. Configure the X Series with the standard sample introduction equipment, i.e. a glass concentric nebulizer, glass impact bead spray chamber and a one-piece torch with 1.5mm ID injector tube. A Peltier spray chamber cooling unit is optional. Ensure that the Xi interface cones are fitted. These are standard with the X5 instrument and an option for the X7. They can be identified as follows:

Xi Sampler - 1.1 mm orifice, no nipple, no holes around the flat circumference

Xi Skimmer - Small pointed skimmer mounted in a copper adapter with two screws

Yellow/orange tab peristaltic pump tubes (5.2.6) should be used for sample and internal standard uptake. Connect the liquid output end of the peristaltic pump tubes to the 1.0 mm (OD) barbed fitting screwed into the Y connector. Note that the barbed fitting may require tightening with a pair of grips to ensure a good fluid-tight seal. The mixed output flow should be connected to the nebulizer. See diagram in Appendix 6 for plumbing schematic. A white/white tab peristaltic pump tube (5.2.7) should be connected to the spray chamber drain outlet at one end and to a tube running into a waste vessel at the other and wound on the pump to draw the waste liquid away from the spray chamber.

- 11.1.2. Perform the daily maintenance as outlined in Appendix 3.
- 11.1.3. Switch the instrument into the *Operate* state by clicking the *ON* button at the top of the screen. During the automated ignition sequence, the following processes occur:
- Torch purge with argon gas
  - RF power match
  - Plasma ignition
  - Slide valve open
  - Electronics on

This process takes about two minutes. Upon successful ignition, the software will display *Operate* in the *Instrument State* bar. If the event of unsuccessful ignition, the software will display an error message and/or place a message in the *Technician Event Log*. Upon unsuccessful ignition, inspect the sample introduction equipment and torch, ensuring a good gas-seal at each connection and ensuring the torch is not misaligned or damaged. If all appears satisfactory, the ignition may be attempted again. If the ignition process consistently fails, contact your local Thermo service agent for advice.

- 11.1.4. Once the instrument is in the *Operate* state, it should be left for 30 minutes to reach thermal equilibrium prior to starting analytical measurements. The optimization (tuning), performance testing and instrument set-up calibrations may be performed after 15 minutes. Ensure that the peristaltic pump is operated at a default analytical speed of 15%. This is done by clicking on *Instrument*, *Configurations*, *Configuration Editor*, *View Selected Accessories* (network icon), *Peristaltic Pump*, *Connect* (chain icon). Set pump speed to 15% using the slider bar and adjust the *Settle Time* to 10 seconds and click on *Apply*. Click *OK* to close

the dialogue box.

- 11.1.5. During the initial 15 minutes, the system can be "conditioned" by aspirating the system thoroughly with 2% nitric acid + 1% HCL solution (6.1.4) prior to continuing.
- 11.1.6. Instrument tuning (optimization) is performed using a 20 µg/L Tune Solution (6.4.1), aspirated through the sample uptake tube. Optimization may not be necessary from day to day if the sample introduction system and cones have not been adjusted in any way and if the instrument fulfils the performance requirements given below. If the instrument gives performance exceeding the requirements shown below, proceed to 8.1.7. Otherwise, tune the instrument manually or using *Autotune* while aspirating 20 µg/L Tune Solution (6.4.1) through both the sample and internal standard uptake tubes. *Autotune*, using an appropriately defined sequence is advised (see Appendix 4).

The final conditions must give the following:

<sup>9</sup> Be	>5000cps
<sup>115</sup> In	>50000cps
<sup>208</sup> Pb	>25000cps
<sup>156</sup> CeO/ <sup>140</sup> Ce	<0.02

If the above criteria are met, proceed to 8.1.7. If the above criteria are not met, do not proceed. Check that the tune solution was prepared as per instructions in (6.4.1) and remake if necessary. If the sensitivity is below the minimum requirement, a new detector plateau may be required (see Appendix 6), the cones may require cleaning (see Appendix 8), or the nebuliser or sample uptake lines may have become blocked or may not be properly clamped on the peristaltic pump. If the CeO/Ce ratio is >0.02, the nebulizer gas flow can be reduced and/or the sampling depth increased, obtaining a corresponding reduction in oxide formation. Recheck the above parameters after taking any remedial action.

- 11.1.7. Save the satisfactory instrument settings by clicking on the disk icon on the Tune page. Note that this is not necessary if Autotune has been used, as the instrument settings are saved automatically (unless manual adjustments have been made after autotuning).
- 11.1.8. Set-up the resolution as described in Appendix 5.
- 11.1.9. Perform a cross-calibration (and mass-calibration and detector voltage setup if required) as explained in Appendix 6. Note that retuning may be necessary after performing this routine.
- 11.1.10. Aspirate Tune solution (6.4.1) and run a *Performance Report* (see Appendix 4) to confirm the mass-calibration, resolution, minimum sensitivity and maximum cerium oxide requirement given in (8.1.6) and to verify instrument stability. The performance report acquires five consecutive one minute runs and calculates the percentage relative standard deviation (RSD) of the five measurements for each isotope. The RSD of the elemental analytes in the performance report must be <5%. If the performance report passes, proceed to (8.1.11). If the performance report fails, check:

- a. Liquid uptake tubes for kinks or other damage
- b. Condition and position of the peristaltic pump tubing
- c. Tightness of the peristaltic pump clamp screws (these should be just tight enough to draw liquid through the tube smoothly)
- d. Joints of all sample introduction components, ensuring a good seal
- e. Nebulizer for blockage
- f. Salt deposition on cones

Remedy the above as necessary and repeat the test. Note that retuning may be required if any sample introduction components are adjusted or replaced.

Note: Resolution set-up may require adjustment if the resolution check fails (see Appendix 5). Note that the quadrupole and hexapole bias strongly influence abundance sensitivity (Pole Bias should be kept  $>+4V$  and Hexapole Bias  $<-3V$ ).

If the measured mass position for each mass in the performance report is not within  $\pm 0.1$  amu of the nominal mass position, a new mass-calibration must be performed (see Appendix 6).

## 11.2. Sample Analysis

- 11.2.1. Open the method template by clicking on *Templates* and then <STL PITTSBURGH ICPMS ANALYSIS>. The method template will be opened. This contains all the saved analytical parameters and only the sample list need be amended.
- 11.2.2. Go to *Sample List*. This grid contains all the information about calibration, QC and samples to be run. The calibration and QC concentration information is already stored. Enter all unknown samples into the list in the appropriate order below the existing calibration and QC samples by overwriting the sample label fields. Delete any QC samples that do not apply to the required method. (If sample list changes are to be made permanent to the method, save the method as a *Template*, by going to *File, Save as Template*. Enter a new name to create an amended method, or use the same name to overwrite the current one.)
- 11.2.3. Once all the sample information is added, check the required autosampler positions have been correctly entered. Amend as necessary. To sequentially renumber positions, add the correct position required for the initiation of the sequence and right mouse click on the first correctly numbered cell. A pop-up menu will appear. Select *Renumber autosampler positions* from this. Ensure that all samples have one survey run and 3 main runs and a probe depth of 155mm.
- 11.2.4. Save the experiment run by clicking on the *File* menu, then *Save as*. Enter the required file name, e.g. *enviro090902* and click *Save*.
- 11.2.5. To print the sample list, go to *Reports* and check the *Sample List* box. Click the refresh icon. The sample list will be displayed in a printable format. Press the print icon. Note that this can only be done with PlasmaLab version 2.3 and above.

## 11.3. Loading the Autosampler

- 11.3.1. Pour the required samples into pre-cleaned 15ml polypropylene test tubes (5.1.4).

To avoid contamination, a small amount of the solution to be analyzed can be poured into the tube and then discarded. This will rinse out any residual contamination.

- 11.3.2. Pour blanks, standards and QCs (positioned in rack 0) into pre-cleaned 50ml polypropylene tubes (5.1.5). To avoid contamination, a small amount of the solution to be analyzed can be poured into the tube and then discarded. This will rinse out any residual contamination. Note that **2% nitric acid** (6.1.4) is used as the calibration blank, IBC, and CCB.
- 11.3.3. For the **serial dilution** ("P") sample(s), dispense  $2.00 \pm 0.02$  mL of the original sample into a pre-cleaned 15 mL polypropylene test-tube (5.1.4) and add  $8.00 \pm 0.08$  mL of 2% nitric acid (6.1.4). Mix well. This is a 5-fold dilution.
- 11.3.4. Place the tubes for each sample into the appropriate position in the rack according to the sample list. Note that the autosampler works on a two-dimensional grid position system by rack number (0-4). See Appendix 9 for autosampler position map.

#### 11.4. Initiating Analysis

- 11.4.1. Place the sample probe into the autosampler arm and the internal standard probe into the internal standard solution (6.4.6).
- 11.4.2. Go to *Instrument, Tune* and click on the accessories dialog icon. Click on *Autosampler* and then on the chain icon to connect. The autosampler should initialize. Ensure that the probe is at the correct height by positioning it so that its tip just protrudes through the hole in the bottom of the arm. Click on the *Go to Wash* icon (faucet) to send the probe to the wash station. Ensure that the wash solution is being correctly delivered to the wash station via the peristaltic pump at the rear of the autosampler. Allow at least 2 minutes for the liquid to be delivered to the sample introduction system.
- 11.4.3. Click on the experiment to be run. Click the *Queue* icon and then *Append* and *OK*. The analysis has now been initiated.
- 11.4.4. To monitor the progress of the analysis, right-mouse click on the *MS* icon at the bottom-right of the screen and select *Open Service Window* from the pop-up menu. The Service Window hovers over the current application window until moved or closed and displays the current instrument activity. This window is also used **to stop an analysis** if required. This is done by clicking on the **<sup>x</sup>Q icon**.
- 11.4.5. To view results as they are generated, click on the experiment icon and go to the *Results* tab. Click on the *Refresh* button or the refresh icon (green circular arrows on a page) to calculate the results from the data obtained.
- 11.4.6. To view calibration plots, click on the *Calibration Data* tab. The calibration for each analyte can be viewed by clicking on the required isotope in the *Analyte* box. Each subsequent set of calibrations (calibration block) can be displayed by selecting the required calibration block from the drop-down combo box, e.g. *FQ Block 1*, *FQ Block 2*, etc. FQ denotes a Fully-Quantitative calibration and SQ denotes a Semi-Quantitative calibration, i.e. a response curve generated from the FQ calibrations. The SQ response curve is used to calculate semi-quantitative concentrations if required.
- 11.4.7. To view data, click on the *Numerical Results* tab. The *Analyte Dilution Conc.* tab is

a tabular display of the calculated corrected concentrations for each analyte. These values have been corrected for internal standardization, external drift correction (if used), and dilution (where entered). The *Mass Uncorrected ICPS* tab shows the uncorrected raw data for each measured mass in units of integrated counts per second (ICPS). The *Analyte ICPS* tab shows integrated counts per second data that has been mathematically corrected for blank deduction, internal standardization, drift correction (if used), and dilution (as appropriate). The *Survey* tabs show the data integrated from the survey scan for each sample. Any concentrations displayed in the survey page will be semi-quantitative only.

11.4.8. To edit the amount of data on screen (filter the results display), click on the filter icon (funnel and lightening): Alter the numerical values or the check boxes to select the required data to display and click on *OK*. To jump directly to a particular sample of interest, find the sample in the drop-down combo box at the top of the data display and click on it.

11.4.9. To display mass-spectra, click on the *Spectra* tab. Display the spectrum for a particular sample by double-clicking on the sample name in the selection box on the left of the screen. Note that several spectra may be overlaid by double-clicking on each sample to be displayed. To zoom into a particular area, click the zoom icon (magnifying glass) and click and drag on the spectral display to zoom into the required area. The dashed-lines represent data acquired in the analogue mode of the detector whilst the solid-lines represent pulse-count data. To remove the noise associated with analogue detection at low signal levels, point at the display and right-mouse click to bring up a menu. Go to *View Options* and then click on *Eliminate Analogue Noise*. To identify a peak, click on it and wait for the options for that mass to be displayed in the box above the spectral display. To fingerprint a spectrum, double click on the species to fingerprint in the options box. This will overlay the isotopic pattern for the selected species, based on the lowest relative intensity signal for the pattern masses. The spectra may be navigated by using the arrow buttons above the display. Allow the arrow cursor to hover over each button for an on-screen explanation of its function.

## 11.5. Post-Analysis Data Processing

### 11.5.1. Internal Standards

11.5.1.1. Check the internal standard recovery percentage for each internal standard isotope used for every sample. The percentage for each isotope must be within the range 30-120% for method 6020 and 60 – 125% for method 200.8.

11.5.1.2. If above 120%, check that the other internal standard isotopes show similar deviation. If not, this may be due to the presence of the internal standard element in the sample. This is particularly common with the isotopes of Li, Sc and Y in environmental materials. If this is the case, the affected internal standard isotope may be excluded for the sample affected, as follows. Go to the *Sample List*.

Find the sample affected and select it in the list by clicking on the box in the left-hand column. Click *Show Advanced* and go to *Internal Standards*. Click on *New Internal Standard Set*. Select the affected isotope(s) in the *Internal Standards* box on the right. Remove the affected isotope from the *Internal Standards* box by using the left hand

arrow button (<<). Recalculate the results for this sample by going back to *Results* and clicking on *Refresh*.

- 11.5.1.3. If any internal standard isotope is outside the range 30-120% and all other internal standard isotopes show similar values for that sample, the instrument may have drifted, or the sample may be producing a suppression or enhancement effect. Find the nearest blank following the sample in question and check its internal standard results. If these are similarly reduced or elevated, the instrument has drifted and the samples must be reanalyzed from the last compliant blank. If the blank does not exhibit similar drift, the sample must be producing a suppression or enhancement effect due to its matrix. In this case the sample must be re-analyzed after a five-fold (1+4) or a two-fold (1+1) dilution to reduce the matrix effect.

#### 11.6. General protocols

- 11.6.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.
- 11.6.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.
- 11.6.3. An analytical run will consist of all customer samples and quality control samples analyzed under a daily initial calibration. Each new initial calibration will begin a new analytical run.
- 11.6.4. Type in the QC and sample information into the autosampler table.
- 11.6.5. In order to use the ICP-MS data upload program into LIMS, the following naming conventions must be followed:
- Samples are identified by the 5 character work order number
  - Matrix spikes, duplicates, and matrix spike duplicates are identified by the 5-character work order number followed by S (matrix spike), D (matrix spike duplicate) or X (sample duplicate).
  - Prep Blanks are identified by the 5 character work order number followed by B.
  - LCSs are identified by the 5 character work order number followed by C (LCS) or L (LCS Duplicate).

#### 11.7. Initial Calibration

- 11.7.1. Open a new dataset using the date and instrument in the title. For instance the first run (A) on instrument 2 on JAN 1, 2003 would be X30101A.
- 11.7.2. Open the appropriate method if one already exists or create a new one for the analytes to be quantitated in the run. Solicit the assistance of a senior ICP-MS operator in creating a new method.

- 11.7.3. See Tables 7, 8, and 9 for recommended isotopes and interference equations for commonly analyzed elements.
  - 11.7.4. If no recommended isotopes are given for the element to be analyzed, consult a senior ICP-MS operator or appropriate reference (see Section 13.2).
  - 11.7.5. See Table 10 for commonly used internal standards.
  - 11.7.6. All masses which could affect data quality should be monitored to determine potential interferences either simultaneously during an analytical run or in a separate scan.
  - 11.7.7. Internal standards are added to all standards and samples by the instrument prior to analysis.
  - 11.7.8. Use of an existing autosampler table is suggested. A read delay of 45 to 60 seconds is used between all analyses.
  - 11.7.9. Calibration consists of a blank and a single calibration standard (STD1, see Table 2 for concentrations) in accordance with the manufacturer's procedure. Use the average of three integrations for both calibration and sample analyses.
- 11.8. The order of analysis for the initial QC samples and calibration should be:
- 1. Rinse
  - 2. Performance Report (Tune Check)
  - 3. STD1 (Calibration Standard)
  - 4. STD2 (2x Calibration Standard)
  - 5. STD3 (3X Calibration Standard)
  - 6. ICV (Second source, must be  $\pm 10\%$  of true value)
  - 7. ICB
  - 8. CRI / RLV (Reporting Limit Verification Standard)
  - 9. ICSA (Interference check solution.)
  - 10. ICSAB (Interference check solution,  $\pm 20\%$  of true value)
  - 11. CCV
  - 12. CCB
  - 13. Prep QC such as LCS or MB, followed by samples (up to 10 runs)
- 11.8.1. To continue the analytical run, add an additional 10 runs followed by CCV/CCB, and repeat for up to 24 hours.
- 11.8.2. Analysis sequence when out-of-control QC is observed: Recalibrate and rerun all affected samples (including initial QC)

## 12. DATA ANALYSIS AND CALCULATIONS

- 12.1. All pertinent calculations are performed by the ELAN software.

12.2. Reporting Requirements

- 12.2.1. Units are ug/L for aqueous samples and mg/kg for soil samples.
- 12.2.2. If dilutions were required due to insufficient sample, interferences, or other problems, the laboratory reporting limits are multiplied by the dilution factor.
- 12.2.3. For results less than 10 mg/L, two significant figures will be reported. For results greater than or equal to 10 mg/L, three significant figures will be reported. Refer to Policy QA-004 for additional information on significant figures and rounding.
- 12.2.4. Document any non-standard procedures or anomalies by using the anomaly program (Clouseau).

12.3. Data Package Requirements

- 12.3.1. A complete data package consists of: the daily tuning package, the method printout, run log, internal standard summary for 5.2 only, standards documentation, level 1 checklist, and all raw data.
- 12.3.2. Level I review will be completed by the analyst.
- 12.3.3. Level II review will be completed by a senior level laboratory analyst familiar with the technical aspects of ICP-MS and in accordance with the ICP-MS DATA REVIEW checklists. The instrument operator of an analytical run may not perform the Level II review for that run.

12.4. Disk Back-Up

- 12.4.1. Datasets must be backed up monthly onto CD disks. All the datasets for each calendar month are copied onto a disk. Note that the dataset names do not change. The Optimization ("optimization") Reprocess files for that month are also copied onto the disk. The disks are stored in a storage cabinet in the laboratory for 5 years from the last day of the month saved.
- 12.4.2. Laboratory instrument data archival will be performed entirely on network servers as new hardware is available. Full implementation is expected by the end of calendar year 2002.

**13. METHOD PERFORMANCE**

13.1. Initial Demonstration of Capacity

Prior to analysis of any analyte using Method 6020, the following requirements must be met.

- 13.2. Instrumentation Detection Limit (IDL) – IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined quarterly for method 6020 for the standard analytes listed in Appendix A. For method 200.8 IDLs will be determined annually. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined.

- 13.2.1. For 6020 the IDLs shall be determined by performing a blank analysis on 3 non-consecutive days with 7 consecutive measurements per day. The IDL is calculated by summing the standard deviations of the measurements from each day. For 200.8 the IDL is determined by performing 10 replicate blank analysis and multiplying the resulting standard deviation by 3.
- 13.2.2. Each measurement must be performed as though it were a separate analytical

sample.

- 13.2.3. Each measurement must be followed by a rinse and/or any other procedure normally performed between the analyses of separate samples.
- 13.2.4. The IDL measurement must consist of the same number of replicates used for analytical samples with the average result used for reporting.
- 13.3. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. MDL's must be redetermined on an annual basis as detailed in Policy S-Q-003 and further defined in QA-005.
  - 13.3.1. On occasion, a non-routine analyte is requested by the client. In lieu of a full MDL study, a standard containing the non-routine analyte must be analyzed. The concentration of the standard must correspond to the reporting limit or  $\frac{1}{2}$  the reporting limit. This is to verify that the method can satisfactorily quantify the element near the chosen reporting limit. The recovery of the standard must be between 50% and 150% of the expected value. The standard analysis should be kept with the analytical data.
- 13.4. Linear Range Verification (LR) - The linear range is determined semi annually (2x/year) for each element on the standard list. Some regulatory programs, such as AFCEE, may require more frequent determinations.
  - 13.4.1. To determine the linear range, analyze 3 standards at increasing concentration up to 90% of the last concentration where the element was within 10 % of true value is considered the upper linear range.
  - 13.4.2. An alternative is to prepare a higher concentration standard and run this in the analytical run. If this standard is within 10% of the expected value this value can be used as the upper linear range. If this option is chosen, then note the action in an anomaly.
- 13.5. Training Qualification
  - 13.5.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

## 14. POLLUTION PREVENTION

- 14.1. Standards or solutions are not approved for disposal to the sink.

## 15. WASTE MANAGEMENT

- 15.1. Waste generated in the procedure must be segregated and disposed in accordance with the STL Corporate Safety Manual and facility hazardous waste procedures. Contact the Environmental Health and Safety Coordinator or the Hazardous Material Technician with questions regarding disposal.
- 15.2. Samples and other solutions containing high concentrations of toxic materials must be segregated and disposed in accordance with the STL Corporate Safety Manual and facility hazardous waste procedures. Contact the Environmental Health and Safety Coordinator or the Hazardous Material Technician with questions regarding disposal.
- 15.3. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.
- 15.4. Expired standards must be rotated out of the laboratory to the Hazardous Waste disposal

area.

## 16. REFERENCES

- 16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, September, 1994.
- 16.2. Thermo Electron X Series Users Manual
- 16.3. EPA Method 6020 CLP M, Version 8.
- 16.4. Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111) ; Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma - Mass Spectrometry, Revision 5.4, 1994
- 16.5. EPA Method 200.8 EMSL office of Research & Development, Cincinnati, OH (Draft Method, Revision 4.3, August 1990).

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## 17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

### Appendices

- |             |  |
|-------------|--|
| Appendix 1  | Cleaning Procedure for Glass- and Plastic-ware                 |
| Appendix 2  | Wash Solution Preparation Instructions                         |
| Appendix 3  | Daily Instrument Maintenance                                   |
| Appendix 4  | Autotune and Performance Reports                               |
| Appendix 5  | Resolution Setup   |
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| Appendix 9  | Autosampler Position Map                                       |
| Appendix 10 | ILM05.2D Contract Required Quantitation Limits (CRQLs)         |
| Appendix 11 | Spiking Levels   |
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| Appendix 14 | Glossary of Abbreviations                                      |

17.1. Tables

TABLE 1						
STANDARD ANALYTE LIST AND REPORTING LIMITS*						
Element	Symbol	CAS #	Aqueous RL mg/L	Aqueous QC SPIKE Mg/L	Soil RL mg/Kg	Soil QC SPIKE Mg/kg
Aluminum	Al	7429-90-5	0.03	2.0	3.0	200
Antimony	Sb	7440-36-0	0.002	0.10	0.2	10
Arsenic	As	7440-38-2	0.002	0.04	0.2	4
Barium	Ba	7440-39-3	0.010	2.0	1.0	200
Beryllium	Be	7440-41-7	0.001	0.05	0.1	5
Boron	B	7440-42-8	0.005	1.0	0.5	100
Cadmium	Cd	7440-43-9	0.001	0.05	0.1	5
Calcium	Ca	7440-70-2	0.10	50	10.0	5000
Chromium	Cr	7440-47-3	0.002	0.2	0.2	20
Cobalt	Co	7440-48-4	0.0005	0.5	0.05	50
Copper	Cu	7440-50-8	0.002	0.25	0.2	25
Iron	Fe	7439-89-6	0.05	1.0	5.0	100
Lead	Pb	7439-92-1	0.001	0.02	0.1	2
Magnesium	Mg	7439-95-4	0.10	50	10.0	5000
Manganese	Mn	7439-96-5	0.0005	0.5	0.05	50
Molybdenum	Mo	7439-98-7	0.005	1.0	0.5	100
Nickel	Ni	7440-02-0	0.002	0.5	0.2	50
Potassium	K	7440-09-7	0.100	50	10.0	5000
Selenium	Se	7782-49-2	0.005	0.01	0.5	1
Silver	Ag	7440-22-4	0.001	0.05	0.1	5
Sodium	Na	7440-23-5	0.10	50	10.0	5000
Strontium	Sr	7440-24-6	0.005	1.0	0.5	100
Tin	Sn	7440-31-5	0.005	2.0	0.5	200
Titanium	Ti	7440-03-26	0.005	1.0	0.5	100
Thallium	Tl	7440-28-0	0.001	0.05	0.1	5
Vanadium	V	7440-62-2	0.001	0.5	0.1	50
Zinc	Zn	7440-66-6	0.005	0.5	0.5	50

\* Note: These are the routine reporting limits for most sample types. Lower reporting limits may be achievable for special projects. Difficult sample matrices may cause reporting limits to be raised.

TABLE 2			
Composition of the CAL Standard			
Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.200	Mn	0.200
Al	1.00	Mo	0.200
As	0.200	Na	100
B	0.200	Ni	0.200
Ba	0.200	Pb	0.200
Be	0.200	Sb	0.200
Ca	100	Se	0.200
Cd	0.200	Si	10
Co	0.200	Sn	0.200
Cr	0.200	Sr	0.200
Cu	0.200	Ti	0.200
Fe	50	Tl	0.200
K	100	V	0.200
Mg	100	Zn	0.200

TABLE 3			
Composition of the ICV Standard			
Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.08	Mn	0.08
Al	0.4	Mo	0.08
As	0.08	Na	40
B	0.08	Ni	0.08
Ba	0.08	Pb	0.08
Be	0.08	Sb	0.08
Ca	40	Se	0.08
Cd	0.08	Si	4.0
Co	0.08	Sn	0.08
Cr	0.08	Sr	0.08
Cu	0.08	Ti	0.08
Fe	20	Tl	0.08
K	40	V	0.08
Mg	40	Zn	0.08

TABLE 4			
Composition of the ICSA Standard			
Element	Concentration ug/mL	Element	Concentration ug/mL
Al	100	P	100
Ca	100	S	100
Fe	100	C	200
K	100	Cl <sup>-</sup>	1000
Mg	100	Mo	2.0
Na	100	Ti	2.0

**TABLE 5**  
**Composition of the ICSAB Standard**

Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.02	Na	100
Al	100	Ni	0.02
As	0.02	Pb	0.02
B	0.05	Sb	0.02
Ba	0.02	Se	0.05
Be	0.02	Si	0.50
Ca	100	Sn	0.10
Cd	0.02	Sr	0.02
Co	0.02	Ti	2.0
Cr	0.02	Tl	0.02
Cu	0.02	V	0.02
Fe	100	Zn	0.025
K	100	P	100
Mg	100.0	S	100
Mn	0.0225	C	200
Mo	2.00	Cl-	720

TABLE 6 <sup>1</sup>					
COMMON MOLECULAR ION INTERFERENCES IN ICP-MS					
Molecular Ion	Mass	Element Interferences <sup>2</sup>	Molecular Ion	Mass	Element Interferences <sup>2</sup>
<b>BACKGROUND MOLECULAR IONS</b>					
NH <sup>+</sup>	15		<sup>38</sup> ArH <sup>+</sup>	39	
OH <sup>+</sup>	17		<sup>40</sup> ArH <sup>+</sup>	41	
OH <sub>2</sub> <sup>+</sup>	18		CO <sub>2</sub> <sup>+</sup>	44	
C <sub>2</sub> <sup>+</sup>	24		CO <sub>2</sub> H <sup>+</sup>	45	Sc
CN <sup>+</sup>	26		ArC <sup>+</sup> , ArO <sup>+</sup>	52	Cr
CO <sup>+</sup>	28		ArN <sup>+</sup>	54	Cr
N <sub>2</sub> <sup>+</sup>	28		ArNH <sup>+</sup>	55	Mn
N <sub>2</sub> H <sup>+</sup>	29		ArO <sup>+</sup>	56	
NO <sup>+</sup>	30		ArOH <sup>+</sup>	57	
NOH <sup>+</sup>	31		<sup>40</sup> Ar <sup>36</sup> Ar <sup>+</sup>	76	Se
O <sub>2</sub> <sup>+</sup>	32		<sup>40</sup> Ar <sup>38</sup> Ar <sup>+</sup>	78	Se
O <sub>2</sub> H <sup>+</sup>	33		<sup>40</sup> Ar <sub>2</sub> <sup>+</sup>	80	Se
<sup>36</sup> ArH <sup>+</sup>	37				
<b>MATRIX MOLECULAR IONS – Chloride</b>					
<sup>35</sup> ClO <sup>+</sup>	51	V	<sup>37</sup> ClOH <sup>+</sup>	54	Cr
<sup>35</sup> ClOH <sup>+</sup>	52	Cr	<sup>35</sup> ClO <sup>+</sup>	51	V
<sup>37</sup> ClO <sup>+</sup>	53	Cr	<sup>35</sup> ClOH <sup>+</sup>	52	Cr
Ar <sup>35</sup> Cl <sup>+</sup>	75	As	Ar <sup>37</sup> Cl <sup>+</sup>	77	Se
<b>MATRIX MOLECULAR IONS – Sulfate</b>					
<sup>32</sup> SO <sup>+</sup>	48		<sup>34</sup> SOH <sup>+</sup>	51	V
<sup>32</sup> SOH <sup>+</sup>	49		SO <sub>2</sub> <sup>+</sup> , S <sub>2</sub> <sup>+</sup>	64	Zn
<sup>34</sup> SO <sup>+</sup>	50	V, Cr			
Ar <sup>32</sup> S <sup>+</sup>	72		Ar <sup>34</sup> S <sup>+</sup>	74	
<b>MATRIX MOLECULAR IONS – Phosphate</b>					
PO <sup>+</sup>	47		PO <sub>2</sub> <sup>+</sup>	63	Cu
POH <sup>+</sup>	48				
ArP <sup>+</sup>	71				
<b>MATRIX MOLECULAR IONS – Group I, II Metals</b>					
ArNa <sup>+</sup>	63	Cu	ArCa <sup>+</sup>	80	
ArK <sup>+</sup>	79				
<b>MATRIX OXIDES<sup>3</sup></b>					
TiO	62-66	Ni, Cu, Zn	MoO	108-116	Cd
ZrO	106-112	Ag, Cd			

<sup>1</sup>From Method 200.8, Section 13.2.6

<sup>2</sup>Method elements or internal standards affected by the molecular ions.

<sup>3</sup>Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes be monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

TABLE 7			
RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED <sup>1</sup>			
Isotope	Element of Interest	Isotope	Element of Interest
<b>27</b>	Aluminum <sup>2</sup>	<b>80,78,82,76,77,74</b>	Selenium
<b>121,123</b>	Antimony <sup>2</sup>	<b>107,109</b>	Silver <sup>2</sup>
<b>75</b>	Arsenic <sup>2</sup>	<b>23</b>	Sodium <sup>2</sup>
<b>138,137,136,135,134,132,130</b>	Barium <sup>2</sup>	<b>203, 205</b>	Thallium <sup>2</sup>
<b>9</b>	Beryllium <sup>2</sup>	<b>51,50</b>	Vanadium <sup>2</sup>
<b>114,112,111,110,113,116,106,108</b>	Cadmium <sup>2</sup>	<b>66, 68</b>	Zinc <sup>2</sup>
<b>42,43,44,46,48</b>	Calcium <sup>2</sup>	<b>83</b>	Krypton
<b>52,53,50,54</b>	Chromium <sup>2</sup>	<b>72</b>	Germanium
<b>59</b>	Cobalt <sup>2</sup>	<b>139</b>	Lanthanum
<b>63,65</b>	Copper <sup>2</sup>	<b>140</b>	Cerium
<b>56,54,57,58</b>	Iron <sup>2</sup>	<b>129</b>	Xenon
<b>206,207,208</b>	Lead <sup>2</sup>	<b>118</b>	Tin
<b>24,25,26</b>	Magnesium <sup>2</sup>	<b>105</b>	Palladium
<b>55</b>	Manganese <sup>2</sup>	<b>47,49</b>	Titanium
<b>98,96,92,97,94</b>	Molybdenum	<b>125</b>	Tellurium
<b>58,60,62,61,64</b>	Nickel <sup>2</sup>	<b>69</b>	Gallium
<b>39</b>	Potassium <sup>2</sup>	<b>35,37</b>	Chlorine

<sup>1</sup> From Method 6020 CLP-M, Table 9

<sup>2</sup> Element approved for ICP-MS determination by SW846 Method 6020 CLP-M

NOTE: Isotopes recommended for analytical determination are **bolded**.

TABLE 8			
RECOMMENDED ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED			
Rare Earth Elements	ICPMS Preferred Mass	Elemental Equations	Additional Masses
Lanthanum	138.906		
Cerium	139.905		
Praseodymium	140.907		
Neodymium	141.908	$-0.125266 * {}^{140}\text{Ce}$	142.910, 144.912
Samarium	151.920	$-0.012780 * {}^{157}\text{Gd}$	144.912
Europium	152.929		
Gadolinium	157.924	$-0.004016 * {}^{163}\text{Dy}$	156.934
Terbium	158.925		
Dysprosium	163.929	$-0.047917 * {}^{166}\text{Er}$	
Holmium	164.930		
Erbium	165.930		
Thulium	168.934		
Ytterbium	173.939	$-0.005935 * {}^{178}\text{Hf}$	171.937
Lutetium	174.941		

TABLE 8			
RECOMMENDED ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED			
Rare Earth Elements			
Other Elements			
Boron	11.009		
Calcium	43.956		
Cesium	132.905		
Galium	68.926		
Germanium	71.922		
Gold	196.967		
Hafnium	177.944		176.944
Holmium	164.930		
Iridium	192.963		
Lithium	7.016		
Tungsten	183.951	-0001242* <sup>189</sup> Os	
Uranium	238.050		
Yttrium	88.905		
Zirconium	238.050		
Niobium	92.906		
Palladium	104.905		
Phosphorus	30.994		
Platinum	194.965		
Rhenium	186.965	-0.099379 * <sup>189</sup> Os	
Rhodium	102.905		
Rubidium	84.912		
Ruthenium	101.904	-0.045678 * <sup>105</sup> Pd	
Scandium	44.956		
Strontium	87.906		
Tantalum	180.948		
Tellurium	127.905	-0.072348 * <sup>129</sup> Xe	
Thorium	232.03		

TABLE 9  
ELEMENTAL EQUATIONS USED TO CALCULATE RESULTS

Element	Elemental Equation	Note
Al	$(1.000) (^{27}\text{C})$	
Sb	$(1.000) (^{121}\text{C})$	
As	$(1.000) (^{75}\text{C}) - (3.1278)[^{77}\text{C}] - (1.0177)(^{76}\text{C})$	Correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.
Ba	$(1.000) (^{137}\text{C})$	
Be	$(1.000) (^9\text{C})$	
Cd	$(1.000) (^{111}\text{C}) - (1.073) [(^{106}\text{C}) - (0.712) (^{106}\text{C})]$	Correction of MoO interference. An additional isobaric elemental correction should be made if palladium is present.
Cr	$(1.000) (^{52}\text{C})$	In 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.
Co	$(1.000) (^{59}\text{C})$	
Cu	$(1.000) (^{63}\text{C})$	
Pb	$(1.000) (^{208}\text{C}) + (1.000) (^{207}\text{C}) + (1.000) (^{206}\text{C})$	Allowance for isotopic variability of lead isotopes.
Mn	$(1.000) (^{55}\text{C})$	
Mo	$(1.000) (^{98}\text{C}) - (0.146) (^{99}\text{C})$	Isobaric elemental correction for ruthenium.
Ni	$(1.000) (^{60}\text{C})$	
Se	$(1.000) (^{82}\text{C})$	Some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.
Ag	$(1.000) (^{107}\text{C})$	
Tl	$(1.000) (^{205}\text{C})$	
Th	$(1.000) (^{232}\text{C})$	
U	$(1.000) (^{238}\text{C})$	
V	$(1.000) (^{51}\text{C}) - (3.127) [(^{53}\text{C}) - (0.113) (^{52}\text{C})]$	Correction of chloride inference with adjustment for Cr53. ClO 51/53 ratio may be determined from the reagent blank.
Zn	$(1.000) (^{66}\text{C})$	
<b>Internal Standards</b>		
Bi	$(1.000) (^{209}\text{C})$	
In	$(1.000) (^{115}\text{C}) - (0.0149) (^{116}\text{C})$	Isobaric elemental correction for tin.
Ge	$(1.000) (^{72}\text{C})$	
Sc	$(1.000) (^{45}\text{C})$	
Tb	$(1.000) (^{159}\text{C})$	
Tm	$(1.000) (^{169}\text{C})$	
Y	$(1.000) (^{89}\text{C})$	

\* Method elements or internal standards affected by the molecular ions.

C = Calibration blank subtracted counts at specified mass.

TABLE 10		
INTERNAL STANDARDS AND LIMITATIONS OF USE		
Internal Standard	Mass	Possible Limitation
Lithium	6	a
Scandium	45	Polyatomic Ion Interference
Germanium	72	
Yttrium	89	a, b
Rhodium	103	
Indium	115	Isobaric Interference by Sn
Terbium	159	
Holmium	165	
Thulium	169	
Lutetium	175	
Bismuth	209	a

a May be present in environmental samples.

b In some instruments Yttrium may form measurable amounts of  $YO^+$  (105 amu) and  $YOH^+$  (106 amu). If this is the case, care should be taken in the use of the cadmium elemental correction equation.

# Appendices

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## **Appendix 1**

### **Cleaning Procedure for Glass- and Plastic-ware**

All glassware and plastic-ware coming into contact with samples, reagents and standards must be cleaned in the following manner. Plastic pipette tips may be cleaned in the same manner by soaking them in a suitable plastic container.

- 1) Completely fill the container to be leached with 10% nitric acid solution (6.1.5) and fit the lid.
- 2) Leave soaking for at least 12 hours.
- 3) Empty the container of acid and rinse thoroughly with laboratory water (6.1.1). Note that the acid may be collected and re-used until it becomes too contaminated.
- 4) Allow the vessel to air-dry in a clean area (preferably Class-1000 or better). If no such clean area is available, the container should be allowed to dry in the cleanest possible environment, or may be emptied of residual water as much as is possible and re-capped.
- 5) Containers should be capped ready for use and stored in the cleanest area available.
- 6) If pre-cleaned containers are to be stored for long periods (weeks to months) prior to use, it is most effective to store them full of laboratory water (6.1.1). This must be discarded and the containers rinsed thoroughly with laboratory water (6.1.1) and dried before use.

## Appendix 2

### Wash Solution Preparation Instructions (2% Nitric Acid (v/v))

A large volume of this solution is required for supply to the autosampler rinse station in order to wash the probe between samples. These instructions detail the preparation procedure for 2.5 L of this solution which is normally sufficient for one day of analytical use. The procedure may be scaled up or down as required.

- 1) Into a 2.5 L container (pre-cleaned as per Appendix 1), add 500±450 mL of laboratory water (6.1.1)
- 2) Add 50±10 mL of concentrated nitric acid (6.1.3)
- 3) Make to 2.50±0.25 L with laboratory water (6.1.1)
- 4) Mix well

#### Notes:

If preparing larger quantities simply scale-up quantities proportionally.

If analyzing Ag, add hydrochloric acid at 1% by adding 50±10 mL of concentrated hydrochloric acid (6.1.2) after step 2.

## **Appendix 3**

### **Daily Instrument Maintenance**

- 1) Wipe all instrument, autosampler and surrounding bench surfaces with a damp wipe – continual cleanliness is important for the minimization of contamination
- 2) Check Wash Solution volume and remake if necessary (see Appendix 2)
- 3) Empty Waste Vessel according to laboratory disposal policy
- 4) Check the condition of all peristaltic pump tubes and replace if required (it is recommended to replace these daily although this may not be necessary with lower sample loads)
- 5) Check condition of sample introduction system and cones and clean and/or replace as necessary (see Appendix 8)
- 6) Ensure instrument fume-extraction system is operational

## Appendix 4

### Autotune and Performance Reports

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#### Description

*Autotune* is a *PlasmaLab* software tool that allows the X Series to be optimized in a consistent, routine manner, giving reproducible levels of performance and saving the operator time and effort. It works by following a pre-defined sequence, optimizing individual instrument parameters in turn. Default sequences are provided with the software upon installation and a further customized sequence is provided on the CD accompanying this productivity pack.

*Performance Reports* are a *PlasmaLab* software tool that allows the X Series performance to be checked on a daily basis. The *Performance Report* can be set-up to give information about instrument sensitivity, stability, background, oxide species, doubly charged species, mass-calibration validity and peak resolution. Like *Autotune*, the *Performance Report* is user definable but defaults are provided with the software. Customized *Performance Reports* are provided on the CD accompanying this package.

The philosophy of use of these tools is as follows. After the sample introduction system or the cones have been removed and replaced or upon using the instrument for the first time or following major adjustments, the full *Autotune* sequence should be used to properly optimize the system. This takes about 15 minutes. From this, an *Autotune Update* sequence can be automatically created. This is a shortened version of the optimization sequence and will take about 5 minutes to run. The performance of the X Series is, in general, very stable from day-to-day, meaning that large amounts of optimization are not normally needed on a daily basis. To check whether optimization is needed, a *Performance Report* can be run initially. The results of this tell the operator if the system requires resolution adjustment, re-mass-calibration, or re-optimization. If the required sensitivity, background, stability or oxide performance is not satisfied, an *Autotune* should be run (the faster *Autotune Update* is normally sufficient). The *Performance Report* should then be repeated to ensure that the problem has been resolved.

#### Installing the EPA Autotune Sequence

To install the custom Autotune sequence, follow the instructions below:

- 1) Insert the CD in the CD ROM drive of the instrument operating PC. Wait for it to autorun and install the Productivity Pack by following the prompts after clicking on *Install*.
- 2) Ensure that PlasmaLab version 2.2 (or higher) has been installed

- 3) In PlasmaLab, go to *Instrument, Tune* and click on the down arrow button next to the *Autotune* icon (musical note).
  - 4) Point to *Tools* in the menu and then select *Import Autotune Sequences*
  - 5) Click *Next* in the Autotune Wizard
  - 6) Click on *Browse* and find the path  
*C:/Program Files/ThermoElemental/PlasmaLab/Data*
  - 7) Select *EPA Autotune Sequence* and click on *Open*
  - 8) Click on *Next*
  - 9) Select *EPA – Xi Interface* and click on *Next*
  - 10) Click on *Finish*
- 

### **Installing the EPA Performance Reports**

To install the custom Performance Reports, follow the instructions below:

- 1) Ensure the Pack is installed from the CD as described above
- 2) Ensure that PlasmaLab version 2.2 (or higher) has been installed
- 3) In PlasmaLab, go to *Instrument, Tune* and click on the down arrow button next to the *Performance Report* icon (musical note on page).
- 4) Point to *Tools* in the menu and then select *Import Performance Report*
- 5) Click *Next* in the Performance Report Wizard
- 6) Click on *Browse* and find the path for the CD ROM drive  
*C:/Program Files/ThermoElemental/PlasmaLab/Data*
- 7) Select *EPA 6020 Report* and click on *Open*
- 8) Click on *Next*
- 9) Select *EPA 6020 2.1* and click on *Next*
- 10) Click on *Finish*

To install the second Performance Report, follow instructions 1) to 10) above, selecting the alternative Performance Report name, i.e. *EPA ILM05\_2D Report*.

### **Running Autotune from the Tune Page**

To run an Autotune Sequence follow the instructions below:

- 1) In PlasmaLab go to *Instrument, Tune* and click on the *Autotune* icon (musical note)
- 2) Select *Run an Existing Autotune Sequence* and click on *Next*
- 3) Select the required sequence, e.g. *EPA Xi Interface*, or *EPA Xi Interface – Update* and click on *Next*
- 4) Ensure that the indicated solution is being aspirated (through both probes if on-line internal standard addition is being used) and allow sufficient time for the solution to be transported into the nebuliser
- 5) Click on *Finish*

The selected Autotune sequence will now be run. To monitor its progress, observe the processes indicated at the bottom left of the PlasmaLab screen and open the Service Window (double-click on *MS* icon at the bottom right of the screen). A printable *Autotune Report* is generated at the end of the sequence. To continue, this report must be closed. To access this report upon closure, go to *Instrument, Configurations, Configuration Editor* and point to the appropriate *Instrument Settings* line. Open a pop-up menu by right-clicking and use the *View Tune Report* selection.

## Running a Performance Report from the Tune Page

To run a Performance Report follow the instructions below:

- 1) In PlasmaLab go to *Instrument, Tune* and click on the *Performance Report* icon (musical note on a page)
- 2) Select *Run an Existing Performance Report* and click on *Next*
- 3) Select the required sequence, e.g. *EPA ILM05 / 6020*, or *EPA 6020* and click on *Next*
- 4) Ensure that the indicated solution is being aspirated (through both probes if on-line internal standard addition is being used) and allow sufficient time for the solution to be transported into the nebulizer
- 5) Click on *Finish*

The selected *Performance Report* will now be run. To monitor its progress, open the Service Window (double-click on *MS* icon at the bottom right of the screen). A printable *Performance Report* is generated at the end of the sequence. To access this report upon closure, go to *Instrument, Tune*, and click on the down arrow to the right of the *Performance Report* icon. Point at *Tools* and then select *View Performance Report Results*. Select the required Performance Report to view and click *OK*.

## Running Performance Reports and Autotune in an Experiment

It is also possible to automate the running of these procedures using an instrument setup sample within an experiment. To do this, insert an *Instrument Setup Sample* at the beginning of the Sample List by selecting the first sample and using a right-mouse-click menu to *Insert New Before*. Define the *Sample Type* for this new sample as *Instrument Setup* and click on *Show Advanced*. Click on the *Instrument Performance Tests* tab and setup the Performance Report and Autotune functions following the logic and using the drop-down combo boxes to select the next action. An example would be as follows:

Acquire Performance Report	<i>EPA ILM05.2 / 6020</i>
If mass calibration verification fails then	<i>Abort the Queue</i>
If the Performance Report fails then	<i>Autotune using EPA – Xi Interface</i>
If the Autotune fails then	<i>Abort the experiment</i>
If the Autotune passes then	<i>re-run the Performance Report</i>
If the Performance Report fails again then	<i>Abort the Queue</i>

When Performance Reports and Autotunes are acquired in this way, the results are stored as part of the experiment report. Note that since this method of acquiring the report is done using the autosampler, the solution concentration should be adjusted if on-line internal standard addition is to be used, e.g. if the addition dilutes the samples 1:1, the solution concentration should be doubled to get an accurate measure of sensitivity.

## Appendix 5

### Resolution Set up

With the instrument in *Operate* mode, aspirate 10 µg/L Tune solution (6.4.1) (through both probes if using on-line internal standard addition). Go to *Instrument, Tune* and stop the real time display (RTD) using the square stop icon. Change the display mode from *Time vs ICPS* to *ICPS on the full mass range*. Insert Be as the mass to monitor and change the spacing to 10, the dwell to 1 ms and the channels to 200. Disable all other masses in the grid. Restart the RTD by clicking on the triangular play icon. The software will display the scanned peak for mass 9, Be. To adjust the resolution, go to the *Global* tab and use the slider bar marked *Standard resolution*. This must be set up to give a peak width of less than 0.75 amu at 5% peak height. This is typically reached at a setting of between 100 and 200. If high resolution mode is to be used, this can be setup by changing the resolution setting on the RTD to *High*. The High Resolution peak width is typically set at about 0.4 amu at 5% peak height, again with values typically between 100 and 200. Note that this method does not use High resolution mode. Each resolution mode should be checked with several other masses across the mass range, typically 55Mn, 115In, 203Tl and 238U are used. Special attention should be paid to the resolution setup for Mn. This is measured at m/z 55, which is adjacent to both iron and argon oxide at mass 56. These high signals must be properly resolved from the low Mn signal in standard resolution mode. When the correct resolution settings are achieved, save the setting using the disk icon. **Note that a new mass-calibration must always be performed after adjustment of the resolution.**

17.2. Appendix 6

17.3. Instrument Calibrations

There are three instrument calibrations that are fundamental for obtaining good quality data on the X Series. These are:

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- 1) Mass-calibration
- 2) Detector Plateau and Analogue voltage set routines
- 3) Detector cross-calibration.

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Mass calibration sets the quadrupole scan parameters to give the correct measured mass positions. The detector plateau sets the optimum voltage on the ion or pulse counting section of the discrete dynode detector. The analogue voltage set routine applies an appropriate voltage on the analogue part of the detector to obtain a cross-calibration factor of approximately 20,000 for a mid-mass isotope. The detector calibration, or cross-calibration, calculates the correction factor, for each measured mass, between the two detector modes, pulse counting and analogue. All three calibrations may be performed in a single routine, or may be performed separately.

### Mass Calibration

A mass-calibration must be performed whenever the resolution settings are adjusted as this will affect the apparent mass position. Mass-calibration must be performed when the Performance Report shows that measured peak positions are >0.1 amu from their nominal position. Mass-calibrations are best performed using a solution containing as many elements as possible or with every analyte required for analysis at the very least. The solution should contain Li and U as these are used as low and high mass datum points. An appropriate concentration solution be used (one that gives between **100,000-1,500,000 cps** for each mass to be calibrated is appropriate). To perform a mass calibration, follow the instructions below.

- 1) Click *Experiment*
- 2) Select *Create New Experiment*
- 3) Click *OK*
- 4) Select the *Default* database
- 5) Click *Open*
- 6) Go to *Sample List*
- 7) Click the *Report* check box in the sample list grid
- 8) Use the drop-down combo box in the *Type* column to select *Instrument Setup*
- 9) Click on the *Show Advanced* button

- 10) Click on the *Instrument Calibrations* tab
- 11) Check the *Mass-Calibration* box
- 12) There is an option to *Update current mass-calibration* or form a *New mass-calibration*. Unless a major hardware change has been performed, the *Update current mass-calibration* option should be selected.
- 13) Click *Queue*
- 14) Save the experiment with an appropriate name, e.g. *masscal 090902* and click *Save*
- 15) Click *Append*
- 16) Click *OK*

Mass-calibration will now be performed.

To view the mass-calibration results, go to *Instrument, Calibrations, Mass-Calibration*. A mass-calibration for each of the two resolution modes is displayed in the graph of Peak Width and Error (y) versus Mass (x). The current mass-calibration is indicated by the row(s) displayed in green. To display alternative mass-calibrations, click on the appropriate date/time-stamped line in the top grid. The Performance Report function can be used to check mass-calibration accuracy (see Appendix 4).

### Detector Plateau and Analogue Voltage Set

These routines can be performed separately, but it is advised to run them simultaneously as described here. The necessary frequency of these calibrations depends upon the amount of signal the detector is exposed to, i.e. how many samples are analyzed, which analytes and what concentrations. For most laboratories running a moderate sample load, this procedure may be run weekly. Up to three masses may be used in this procedure, however here, the use of a single mass is described. A solution that gives a countrate of between **100,000-1,500,000 cps** is appropriate. The default mass used here is indium (m/z 115), so this must be present in the solution for the routine to work. For an X5 instrument, an appropriate concentration would typically be between 10 and 100 µg/L, depending upon the sensitivity of the system. To perform this routine, follow the instructions below.

- 1) Click *Experiment*
- 2) Select *Create New Experiment*
- 3) Click *OK*
- 4) Select the *Default* database
- 5) Click *Open*
- 6) Go to *Sample List*
- 7) Click in the *Report* check box in the sample list grid
- 8) Use the drop-down combo box in the *Type* column to select *Instrument Setup*
- 9) Click on the *Show Advanced* button

10) Click on the *Instrument Calibrations* tab

11) Check the *Set analogue voltage* box

12) Set the *Number of iterations* to 2

13) Click *Queue*

14) Save the experiment with an appropriate name, e.g. *plateau 090902* and click *Save*

15) Click *Append*

16) Click *OK*

The voltage setup will now be performed. To view the plateau, go to *Instrument, Calibrations, Detector Plateau*. A graph of signal intensity (y) versus voltage (x) is displayed. The “knee” inflexion on this plot corresponds to the plateau voltage. This is automatically selected and applied to the detector by the software.

### Detector Calibration (Cross-Calibration)

**This routine must be performed whenever the detector voltages are altered and daily prior to analysis of samples.** The solution used must contain all the analytes to be measured as an absolute minimum. The more analytes present, the better. All analytes should ideally be set at a concentration that gives between **500,000 and 1,500,000cps**. To perform the detector calibration, follow the instructions below:

- 1) Click *Experiment*
- 2) Select *Create New Experiment*
- 3) Click *OK*
- 4) Select the *Default* database
- 5) Click *Open*
- 6) Go to *Sample List*
- 7) Click in the *Report* check box in the sample list grid
- 8) Use the drop-down combo box in the *Type* column to select *Instrument Setup*
- 9) Click on the *Show Advanced* button
- 10) Click on the *Instrument Calibrations* tab
- 11) Check the *Detector Calibrate* box
- 12) Click *Queue*
- 13) Save the experiment with an appropriate name, e.g. *xcal 090902* and click *Save*
- 14) Click *Append*
- 15) Click *OK*

The detector calibration will now be performed. To view the cross-calibration graph, go to *Instrument, Calibrations, Detector Cross-Calibration*. A graph of cross-calibration factor (y) versus mass (x) is displayed. **Use the data table to check that all analytical masses of interest have been used in the cross-calibration.** If not, the cross-calibration factor will be estimated from the equation of the graph. This may result in error.

### All Routines in One

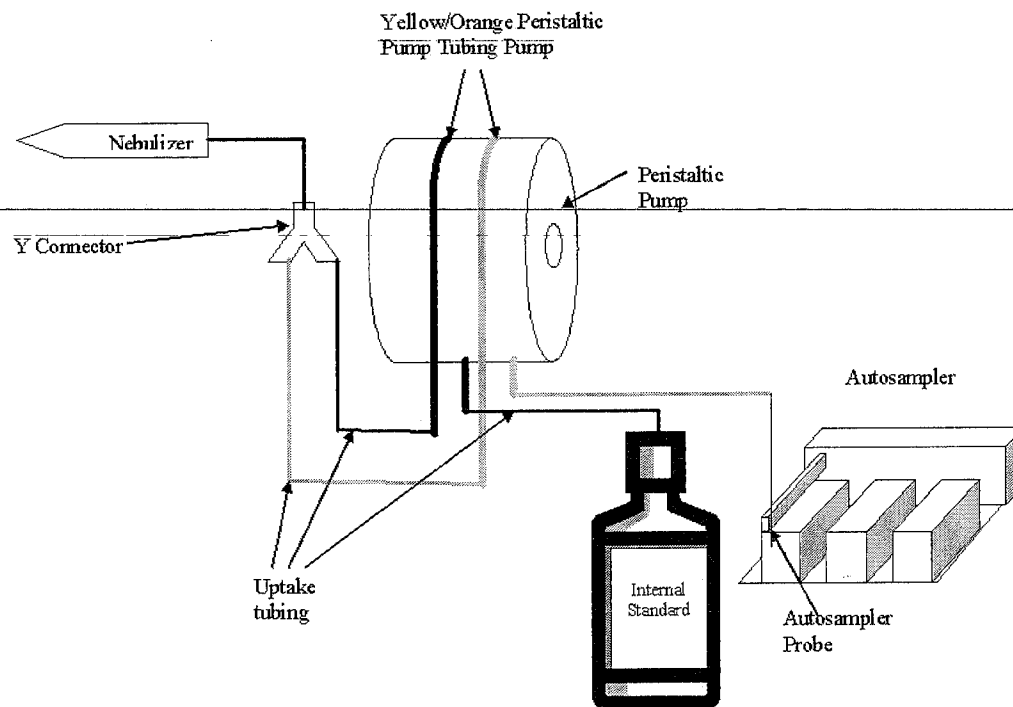
It is possible to run all three of the above routines on a single run if the solution used conforms to all of the criteria spelt out above. To do this, follow the instructions below.

- 1) Click *Experiment*
- 2) Select *Create New Experiment*
- 3) Click *OK*
- 4) Select the *Default* database
- 5) Click *Open*
- 6) Go to *Sample List*
- 7) Click in the *Report* check box in the sample list grid
- 8) Use the drop-down combo box in the *Type* column to select *Instrument Setup*
- 9) Click on the *Show Advanced* button
- 10) Click on the *Instrument Calibrations* tab
- 11) Check the *Mass calibration*, *Detector Calibrate* and *Set analogue voltage* boxes
- 12) Set the *Number of iterations* to 2
- 13) Click *Queue*
- 14) Save the experiment with an appropriate name, e.g. *instr cal 090902* and click *Save*
- 15) Click *Append*
- 16) Click *OK*

The instrument calibrations will now be performed. Each parameter can be viewed as described above.

## Appendix 7

### Sample Introduction Plumbing Diagram



## Appendix 8

### Procedure for Cleaning Sample Introduction Equipment and Cones

- 1) Ensure that the instrument is in the *vacuum* or *shutdown* state (i.e. the plasma is OFF and the slide valve is SHUT)
- 2) Dismantle the sample introduction system as follows:
  - a) Remove the gas connection from the nebulizer
  - b) Remove the sample input plug from the nebulizer
  - c) Remove the metal clip on the spray chamber to elbow joint
  - d) ~~Remove the drain plug from the spray chamber~~
  - e) Slide the spray chamber and nebulizer away from the elbow
  - f) Carefully slide the nebulizer out of the spray chamber and set both pieces aside in a safe place
  - g) Open the torch box and the internal Faraday cage
  - h) Pull the gas connections away from the torch
  - i) Undo the torch catch
  - j) Remove the metal clip on the elbow to torch joint
  - k) Carefully remove the torch from the load coil and set aside in a safe place
  - l) Remove the elbow by sliding it out of the torch box bulkhead toward spray chamber end
  - m) Slide the torch box away from the mass spectrometer to reveal the interface
  - n) Use the flat metal cone tool to undo the locking ring over the sample cone
  - o) Carefully remove the sample cone and set aside in a safe place
  - p) Carefully unscrew and remove the skimmer cone from the interface using the cylindrical aluminium tool and set aside in a safe place
- 3) Clean the cones as follows.
  - a) Carefully place the cones into a large beaker and fill with sufficient 0.05% nitric acid to cover – CAUTION: Stronger acids will corrode the cone material and reduce lifetime
  - b) Place the beaker in an ultrasonic bath for about 10 minutes or until surface deposition has been removed
  - c) Carefully remove the cones from the solution and rinse thoroughly with deionised water
  - d) Allow the cones to air-dry prior to refitting
- 4) Clean the sample introduction equipment as follows.

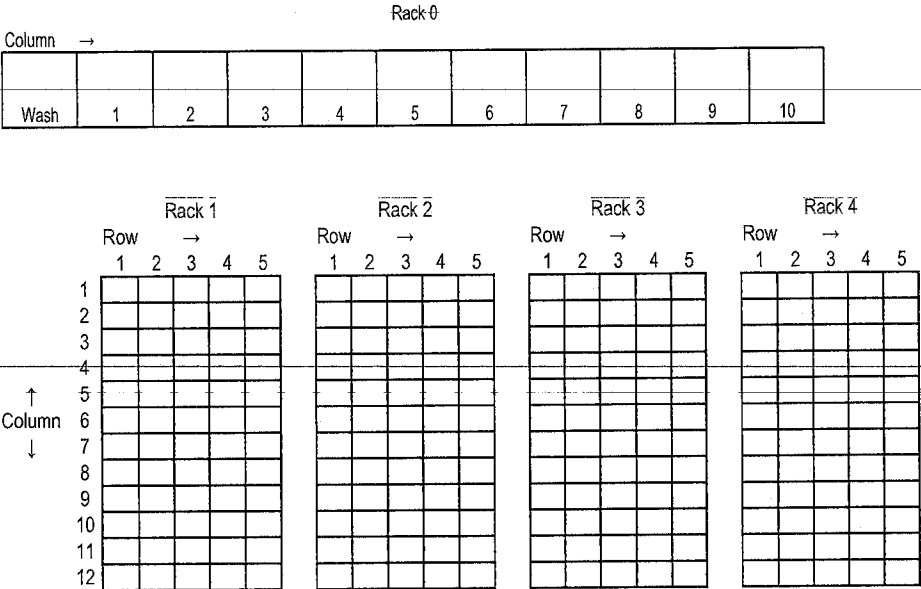
- e) Carefully place the glass sample introduction components into a large beaker and fill with sufficient 10% nitric acid to cover all components
  - f) Place in an ultrasonic bath for between 20 minutes and 1 hour
  - g) Carefully remove the glass components and rinse thoroughly with deionised water
  - h) Allow to air-dry prior to refitting
- 
- 5) Reassemble the components in the reverse order to disassembly

**Note:** Occasionally, glass sample introduction components crack when the ultrasonic cleaning procedure is used. To avoid this, the components may be soaked in acid, as above, for 12 hours, without ultrasonic treatment.

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Thermo Electron can not take any responsibility for any breakage that occurs during cleaning.

Appendix 9  
Autosampler Position Map



NB: This map is only applicable for CETAC ASX-500/510 autosamplers fitted with 60 position racks.

## Appendix 10

### ILM05.2D Contract Required Quantitation Limits (CRQLs)

Analyte	CRQL (µg/L)
Al	30
Sb	2
As	1
Ba	10
Be	1
Cd	1
Ca	(100)
Cr	2
Co	0.5
Cu	2
Fe	(50)
Pb	1
Mg	(100)
Mn	0.5
Ni	1
K	(100)
Se	5
Ag	1
Na	(100)
Tl	1
V	1
Zn	1

CRQLs given in parentheses are not specified for ICP-MS in EPA document ILM05.2 and are for ICP-AES. This is for information only.

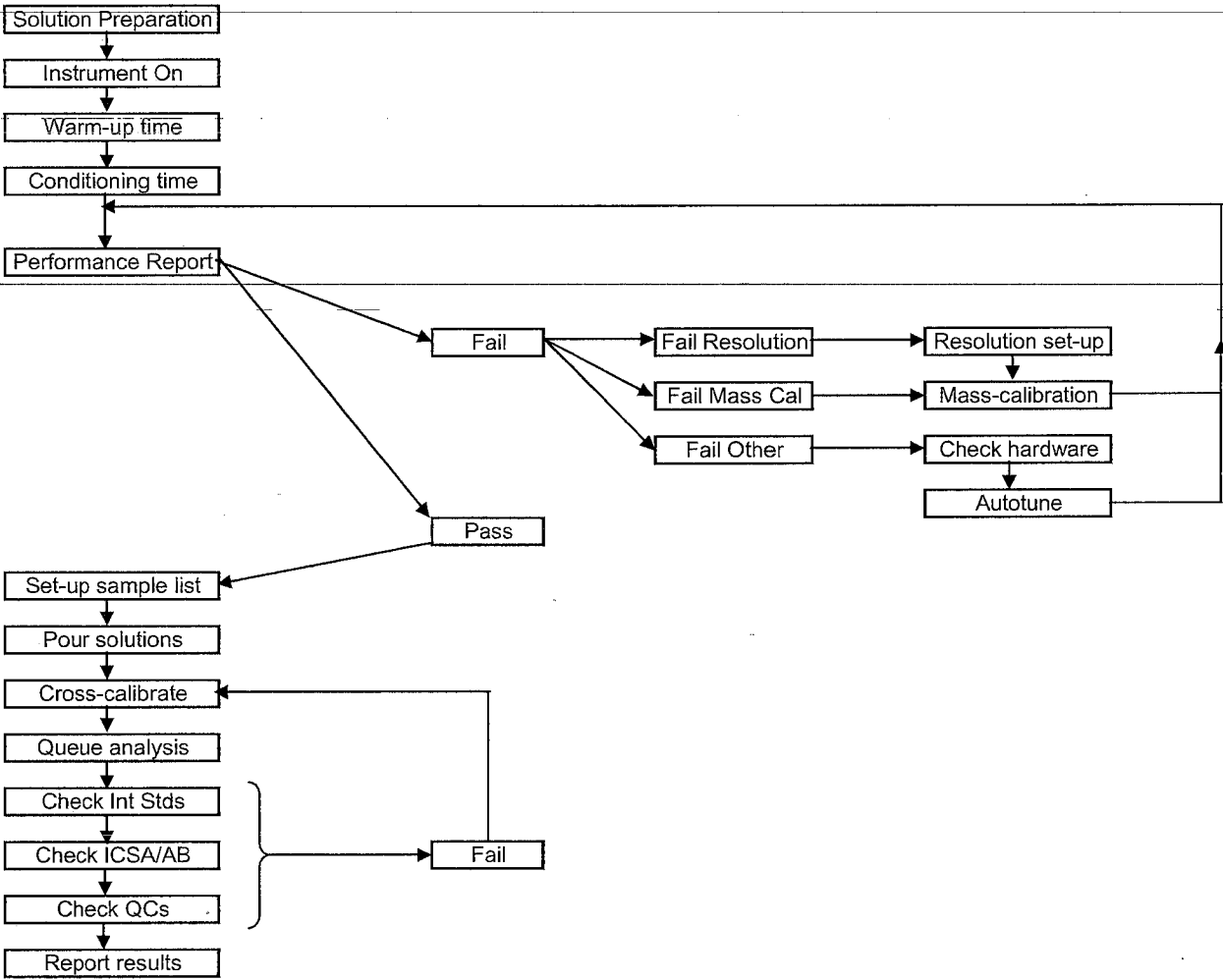
## Appendix 11

### Spiking Levels

(Concentration in Final Solution Based on Instructions Within this Document)

Analyte	Spike Value (µg/L)
Al	2000
Sb	100
As	40
Ba	2000
Be	50
Cd	50
Cr	200
Co	500
Cu	250
Pb	20
Mn	500
Ni	500
Se	10
Ag	50
Tl	50
V	500
Zn	500

Appendix 12  
Work Flow-Chart



## Appendix 13

### Glossary of Abbreviations

QC Code	QC Name	Purpose	Frequency	Limits
ICV	Initial Calibration Verification	checks the calibration against a second calibration source	After initial calibration	90-110%
ICB	Initial Calibration Blank	initial check of read-back at blank level	After initial calibration	<CRQL
CRI	Contract Required Quantitation Limit Check	checks accuracy at the required limit of quantitation	After each calibration and every 20 samples	70-130% 50-150% for Co, Mn, Zn
ICSA	Interference Check Solution A	checks for freedom from interference	After initial calibration	$\pm 3\text{CRQL}$ or $\pm 20\%$ of the true value (whichever is the greater)
ICSAB	Interference Check Solution AB	checks that analytes are accurately measured in an interference-producing matrix	After initial calibration	80-120% of true value
CCV	Continuing Calibration Verification	a continuing periodic check on accuracy and drift	After each calibration and every 10 samples	90-110%
CCB	Continuing Calibration Blank	a continuing periodic check on the read-back at blank levels	After each calibration and every 10 samples	<CRQL
PDS	Post Digestion	checks the	Once every 20	75-125%

QC Code	QC Name	Purpose	Frequency	Limits
	Spike	recovery of analytes spiked into an unknown sample after preparation (digestion)	samples per matrix	
DUP	Duplicate	checks the reproducibility of results by analyzing an unknown sample in duplicate	Once every 20 samples per matrix	±20% Relative Percentage Difference (RPD)
SER	Serial Dilution	checks for matrix effects by assessing the variation of results for an unknown sample before and after dilution	Once every 20 samples per matrix	±10% of the original undiluted result after dilution correction
LCS	Laboratory Control Sample	checks the accuracy of the entire analytical process	Once every 20 samples per matrix	80-120%

## **APPENDIX 45**

Controlled Copy

Copy No.: \_\_\_\_\_

Implementation Date: \_\_\_\_\_

**OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE*****TITLE: Hardness, Total (mg/L as CaCO<sub>3</sub>) by Method 130.2/Method 2340C; and Calcium Hardness or Total Calcium by Method 215.2*****(SUPERSEDES: SOP No. PITT-WC-0004, Revision 3, 1/24/01)**Prepared by: Brenda Benner 4/29/05Reviewed by: Michael T. Wasolowski 4-29-05  
Technical SpecialistApproved by: [Signature] 4-29-05  
Quality Assurance ManagerApproved by: [Signature] for Bill Gehl 4-29-05  
Environment, Health and Safety CoordinatorApproved by: [Signature] 4-29-05  
Laboratory Director

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## 1. SCOPE AND APPLICATION

- 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
- 1.2 Depending on the indicator used, this method can be used to determine either total hardness or calcium hardness, both expressed in mg/L of  $\text{CaCO}_3$ .

## 2. SUMMARY OF METHOD

- 2.1 Total hardness is defined as the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate in mg/L. Calcium hardness is defined as calcium concentration expressed as calcium carbonate in mg/L.
- 2.2 For total hardness, calcium and magnesium ions in the sample are sequestered on addition of disodium ethylenediamine tetraacetate ( $\text{Na}_2$  EDTA). The end point of the reaction is detected by Eriochrome Black T, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered. For calcium hardness or total calcium, calcium ion is sequestered in the same manner, but the titration end point is detected by means of an indicator which combines with calcium only.
- 2.3 The reporting limit for undigested samples is 5 mg/L of  $\text{CaCO}_3$ .
- 2.4 The reporting limit for digested samples is 10 mg/L of  $\text{CaCO}_3$ . This is presented for information purposes. STL Pittsburgh recommends that wastewaters be digested, analyzed by ICP and hardness determined by calculation.

## 3. DEFINITIONS

- 3.1 LCS: Laboratory Control Sample is processed through all method steps with the associated samples. The LCS is used to monitor the accuracy of the analytical process independent of possible interference effects due to sample matrix. Successful analyte recovery for the LCS provides assurance that the method is in control.
- 3.2 LCSD: Laboratory Control Sample Duplicate processed with the LCS when sufficient sample is not available to process a sample duplicate. A LCSD is used to demonstrate batch precision when the client has not supplied sufficient sample to prepare a duplicate sample analysis. A LCSD is required for each batch if a sample duplicate is not present.

- 3.3 MS: Matrix Spike is a replicate portion of one sample in the QC batch that is spiked with a known amount of the target analyte. As a part of the QC batch, it accompanies the sample through all the steps of the analytical process.
- 3.4 MSD: Matrix Spike Duplicate consists of a replicate portion of the sample, which was designated as the MS. This portion is spiked and processed exactly as the MS.
- 3.5 MS/MSD results are used to determine the effects of the sample matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS and the MSD results may not have immediate bearing on any sample except the one spiked.
- 3.6 MB: Method Blank is a control sample that is prepared using reagent water and all other reagents that are used on the associated samples. As part of the QC batch, it accompanies the samples through all steps of the analytical procedure. The method blank is used to monitor laboratory or reagent contamination.
- 3.7 SD : Sample Duplicate is a replicate aliquot of an environmental sample taken from the same sample container, when possible, and processed with the first aliquot of the sample. The sample and sample duplicate results are compared to determine the effect of the sample matrix on the precision of the analytical process. The sample duplicate should be chosen randomly from each batch. The sample should be representative of the entire batch.
- 3.8 QC Batch: The QC batch is a set of 20 or fewer environmental samples plus associated laboratory QC samples that are similar in composition and that are processed within the same time period and with the same reagents and standard lots. Laboratory QC samples such as LCS, matrix QC samples, and blanks are not included in the sample count for QC batching purposes.
- 3.9 Reagent Grade Water: Laboratory water which is produced by a Millipore DI system or equivalent. Reagent grade water must be free of the analyte of interest as demonstrated through the analysis of method blanks.

#### **4. INTERFERENCES**

- 4.1 Excessive amounts of heavy metals can interfere. This is usually overcome by complexing the metals with cyanide. Inhibitors are not necessary for most samples.
- 4.2 For calcium hardness, strontium and barium interfere and alkalinity in excess of 30 mg/L may cause an indistinct end point. Magnesium interference is reduced or

eliminated by raising the pH between 12 and 13 in order to precipitate magnesium hydroxide.

## **5. SAFETY**

- 5.1 Procedures shall be carried out in a manner that protects the health and safety of all STL's associates. The following requirements must be met:
- 5.2 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately.
- 5.3 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory.
- 5.4 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.5 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL's associate. The situation must be reported immediately to a laboratory supervisor.

## **6. EQUIPMENT AND SUPPLIES**

- 6.1 Class "A" burettes, in an appropriate selection of sizes.
- 6.2 Standard laboratory glassware.
- 6.3 Magnetic stir plate and stir bars.
- 6.4 Hot block.
- 6.5 Disposable polypropylene digestion cups.
- 6.6 Polypropylene ribbed watch glasses.

## 7. REAGENT AND STANDARDS

- 7.1 Buffer Solution: Dissolve 1.179g disodium EDTA (analytical reagent grade) and 0.780g  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  (or 0.644g  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ) in 50 mL reagent grade water. Add this solution to a 250mL volumetric flask containing 16.9g  $\text{NH}_4\text{Cl}$  and 143mL conc.  $\text{NH}_4\text{OH}$  with mixing and dilute to volume with reagent grade water. Store in a tightly stoppered plastic bottle to prevent loss of  $\text{NH}_3$  or absorbance of  $\text{CO}_2$ . Discard when 1 or 2mL added to sample fails to produce a pH of  $10.0 \pm 0.1$  at end point of titration. Buffer may also be commercially purchased. Follow manufacturer's expiration date for standard replacement.
- 7.2 Total Hardness Indicator: Calgamite indicator solution: Commercially purchased, follow manufacturer's expiration guidance. If unavailable it may be prepared as follows: 0.10g of dry powder into a 100 ml volumetric flask and dilute to mark w/ reagent water. Alternatively mix together 0.5g Eriochrome Black T and 100g  $\text{NaCl}$ . Store in airtight container. Use the least amount of indicator that provides a sharp end point. Follow manufacturer's expiration date for standard replacement.
- 7.3 Calcium Indicator: Purchased. Follow manufacturer's expiration date for standard replacement.
- 7.4 Standard EDTA titrant, 0.02N: Place 3.723g  $\text{Na}_2\text{EDTA}$  ( $\text{Na}_2\text{H}_2\text{C}_{10}\text{H}_{12}\text{O}_8\text{N}_2 \cdot 2\text{H}_2\text{O}$ ) (analytical reagent grade) in a 1L volumetric flask and dilute to volume with reagent grade water. Check with standard calcium solution (7.5) by titration (10.1). Store in plastic containers, as titrant will extract hardness cations from soft glass. Check standardization semiannually. EDTA titrant can be commercially purchased. Follow manufacturer's expiration date for standard replacement.
- 7.5 Standard calcium solution 0.02 N: This is purchased commercially. If necessary it may be prepared as follows: Place 1.000g anhydrous calcium carbonate (primary standard low in metals) in a 500mL flask. Add slowly 1:1  $\text{HCl}$  (7.6) until all  $\text{CaCO}_3$  has dissolved. Add 200mL of reagent grade water and boil for a few minutes to expel  $\text{CO}_2$ , then cool. Add a few drops of methyl red indicator (7.7) and adjust to intermediate orange color by adding 3N  $\text{NH}_4\text{OH}$  (7.8) or 1:1  $\text{HCl}$ . Transfer to 1L volumetric and dilute to volume with reagent grade water.. Follow manufacturer's expiration date for standard replacement.
- 7.6 Hydrochloric acid solution, 1:1: Add 10 mL of concentrated  $\text{HCl}$  to 10mL reagent grade water in a graduated cylinder. Prepare fresh standard every six months or as needed.
- 7.7 Methyl red indicator: commercially purchased. Follow manufacturer's expiration date for standard replacement.

- 7.8 Ammonium hydroxide solution, 3 N: Dilute 210 mL of conc.  $\text{NH}_4\text{OH}$  to 1L with reagent grade water. Prepare fresh standard every six months or as needed.
- 7.9 Ammonium Hydroxide solution 1N: Dilute 70 mL of concentrated  $\text{NH}_4\text{OH}$  to 1 L with reagent grade water. Prepare fresh standard every six months or as needed.
- 7.10 Concentrated nitric acid ( $\text{HNO}_3$ ).
- 7.11 LCS: 5mL of the calcium solution (see Section 7.5) diluted to 100mL with reagent grade water. This solution has a theoretical value of 50ppm, total hardness and 20ppm total calcium. The LCS should be prepared fresh on each day of use and must be prepared from a second source standard.

## 8. SAMPLE COLLECTION, PREPARATION, AND STORAGE

- 8.1 Samples are acidified to  $\text{pH} < 2$  with  $\text{HNO}_3$ . Holding time is six months from date of sample collection. Plastic or glass containers can be used.

## 9. QUALITY CONTROL

- 9.1 The laboratory control sample is processed with each batch of 20 or fewer environmental samples. The LCS recovery must be  $\pm 20$  percent of the true value. If the LCS fails criteria, the analyst will check calculations and analytical system performance and reanalyze the LCS once. If the LCS is still outside control limits, all samples in the QC batch will be reprepared and reanalyzed. If this is not possible due to limited sample quantity, the laboratory project manager will be notified and an analytical narrative provided with the data. If reparation and reanalysis will be outside of holding time, the client should be notified and approval from the client must be obtained before reanalysis.
- 9.2 Please refer to QA-003 for the selection of any duplicate samples.
- 9.3 A sample duplicate (SD) is analyzed with every set of ten or fewer samples. Acceptance criteria is calculated as relative percent difference (RPD) between the original and duplicate sample analysis and the acceptable range is  $\leq 20$  percent. If the RPD is outside of criteria, the analyst will check calculations and analytical system performance, reanalyze the samples once, evaluate results, and, if appropriate, narrate the problem in the reported data. The duplicate samples are not counted as part of the 20 or fewer environmental samples in the QC batch.

- 9.4 A laboratory control sample duplicate or LCSD is used to demonstrate batch precision when the client has not supplied sufficient sample to prepare a sample duplicate analysis. In this case the LCSD must pass the LCS criteria of  $\pm 20\%$  and the precision criteria of  $\leq 10\%$ . If these criteria are not met, the corrective action noted in section 9.1 would apply.
- 9.5 For programs that require a QC reference sample for every ten samples (ie. such as for NYS samples), an LCS will be analyzed with each batch of 10 or fewer environmental samples.
- 9.6 Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in STL QA Policy: QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL reporting limit.
- 9.7 The method blank (MB) is processed with each batch of 20 or fewer environmental samples. All analyte concentrations in the MB must be less than the reporting limit. If the MB fails criteria, the analyst will check the calculations and analytical system performance and reanalyze the MB once. If the MB is still outside of criteria, all samples associated with the unacceptable blank will be reprepared and reanalyzed. If this is not possible due to limited sample quantity, the laboratory project manager will be notified, and an analytical narrative provided with the data. If reparation and reanalysis is outside of the holding time, the client should be notified and approval from the client must be obtained before reanalysis.

## 10. CALIBRATION AND STANDARDIZATION

- 10.1 Standardization titration procedure: Place 10.0mL standard calcium solution in a flask containing 50mL reagent grade water. Add sufficient buffer to achieve a pH of about 10. Add approximately 1 mL of total hardness (calgamite) indicator. Titrate slowly with stirring until last reddish tinge disappears, adding the last few drops at 3 - 5 second intervals. At the end point, the color is blue. Total titration duration should be 5 minutes from the time of buffer addition.

$$\text{Normality of EDTA} = \frac{0.2 \text{ N}}{\text{mL of EDTA}}$$

Where 0.2 = Normality of Calcium Solution x 10mL of Calcium Solution

## 11. PROCEDURE

### 11.1 Pretreatment:

11.1.1 For drinking, surface, and saline waters (and dilutions of these), no pretreatment or digestion procedure is necessary. Proceed to Section 11.3.

11.1.2 For wastewaters and highly polluted waters, the sample must be digested.

11.2 Digestion procedure: Transfer 100 mL of blank, LCS, sample, or sample duplicate to a disposable polypropylene digestion cup and add 3mL of concentrated  $\text{HNO}_3$ . Cover the beaker with a polypropylene ribbed watch glass. Place the beaker on a hot block and evaporate to near dryness, making certain the sample does not boil. Cool the digestion cup and add another 3 mL of concentrated  $\text{HNO}_3$ . Cover the digestion cup and return to the hot block. Increase the temperature of the hot block so that a gentle reflux occurs. Continue heating. If necessary, add more acid until digestion is complete (normally indicated when digestate is light in color or does not change in appearance with continued refluxing). Set digestion cup aside until cool. Add a small amount (about 3-5mLs) of 1:1 HCl and warm the digestion cup to dissolve any precipitate or residue. Wash down the digestion cup walls and watch glass with reagent grade water and filter the sample to remove silicates and other insoluble material. Adjust the volume to 100 mL with reagent grade water in a volumetric container. Results for samples processed in this manner may be analyzed for "Total Hardness, Total Calcium Hardness, or Total Calcium." Digestion may also be performed using a beaker, a ribbed watch glass, and a hot plate if the hot block is unavailable for use.

11.2.1 With each batch of samples, a prep. blank, LCS and associated batch QC must also be digested. The prep blank is reagent grade water consisting of all the reagents used in the sample.

### 11.3 Titration :

11.3.1 All samples and reagents must be at room temperature. The color change of the indicator is very sluggish in cool temperatures and the indicator decomposes at high temperatures. For all total hardness digestates, as well as non-digested samples having a high-level total hardness concentration (i.e.  $> 5 \text{ mg/L}$ ), proceed to Section 11.3.2. For all non-digested samples with low-level total hardness, proceed to Section 11.3.3. For all calcium hardness or total calcium samples of all concentration levels both digested and non-digested, proceed to Section 11.3.4.

11.3.2 Use 25mL or less LCS, sample, or sample duplicate. Neutralize the pH of the aliquot taken with 3N ammonium hydroxide. Then dilute each treated aliquot

solution to 50mL with reagent grade water. The 50mL aliquot to be titrated should contain about 25mg or less total hardness as  $\text{CaCO}_3$ .

- 11.3.2.1 Add sufficient amount of buffer to achieve a pH of about 10, taking care to watch for any precipitation of  $\text{CaCO}_3$ . The pH should not be so high as to precipitate  $\text{CaCO}_3$ , but needs to be high enough for the indicator to change color. Titration must be completed within 5 minutes of the buffer addition. Samples should require <15 mL EDTA titrant, or a sample dilution is necessary. Add a small scoop of total hardness indicator to the prepared aliquot and titrate slowly with EDTA while constantly stirring the sample. The reddish tint of the indicator will change to a blue end point. Record mL titrant and sample volume used on the bench sheet. Proceed to Section 11.4.
- 11.3.3 For nondigested low-level total hardness determinations, use 100mLs of the sample or blank. Neutralize the pH of the aliquot taken with 1N ammonium hydroxide.
- 11.3.3.1 For each nondigested sample or blank use two scoops of total hardness indicator and enough buffer to achieve a pH of about 10, taking care to watch for any precipitation of  $\text{CaCO}_3$ . The pH should not be so high as to precipitate  $\text{CaCO}_3$ , but needs to be high enough for the indicator to change color. Titration must be completed within 5 minutes of buffer addition. Titrate the EDTA titrant slowly while constantly stirring the sample. The reddish tint of the indicator will change to a blue end point. Record mL of titrant and sample volume used on the bench sheet. Proceed to Section 11.4.
- 11.3.4 For calcium hardness or total calcium determinations (of all concentration level samples and every digestate for calcium hardness and total calcium), use 50mL (or an aliquot diluted to 50mL) of sample, blank, LCS, or sample duplicate. Adjust the pH to 12 to 13 with 3N ammonium hydroxide solution. The 50mL aliquot to be titrated should contain 5 to 10mg total calcium or about 25mg or less calcium hardness as  $\text{CaCO}_3$ .

NOTE: If the alkalinity is >300mg/L  $\text{CaCO}_3$  and cannot be reduced by dilution because of low calcium concentration, then the alkalinity must be decreased by acidifying the sample, boiling one minute, and cooling before the 50mL aliquot can be taken.

11.3.4.1 Add approximately 1 ml of calcium indicator and immediately titrate with EDTA while continuously stirring. Record mL of titrant and sample volume used on the benchsheet. Proceed to Section 11.4.

11.4 Any authorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

## 12. DATA ANALYSIS AND CALCULATIONS

12.1 Hardness (EDTA)

$$\text{mg CaCO}_3 / \text{L} = \frac{A \times N \times 50,000}{\text{mL sample}}$$

Where:

A = mL EDTA titrant used.

N= normality of EDTA titrant.

12.2 Total calcium:

$$\text{mg / L Ca} = \frac{A \times N \times 20,040}{\text{mL of sample}}$$

Where:

A and N are as defined as in 12.1

12.3 Calcium hardness :

$$\text{mg / L CaCO}_3 = \frac{A \times N \times 50,000}{\text{mL of sample}}$$

Where:

A and N are defined as in 12.1

12.4 Duplicate sample (Relative Percent Difference) :

$$RPD = \frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2}\right)} \times 100 \%$$

X<sub>1</sub> = Original Result

X<sub>2</sub> = Duplicate Result

12.5 LCS Percent Recovery:

$$\text{LCS \% Recovery} = \left( \frac{\text{Observed Conc. in LCS}}{\text{True LCS Conc.}} \right) \times 100\%$$

### 13. METHOD PERFORMANCE

13.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. The group/team leader must document the training and PE performance and submit the results to the QA Manager for inclusion in associate training files.

### 14. POLLUTION PREVENTION

14.1 This method does not contain any specific modifications that serve to minimize or prevent pollution.

### 15. WASTE MANAGEMENT

15.1 Waste generated in the procedure will be segregated, and disposed according to the facility hazardous waste procedures. The Health and Safety Coordinator should be contacted if additional information is required.

### 16. REFERENCES

- 16.1 Method 130.2, Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020; March 1983.
- 16.2 Method 215.2, Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020; March 1983.
- 16.3 Method 2340C, Standard Methods for the Examination of Water and Wastewater, 18th Ed., 1992.
- 16.4 QA-003, STL QA Program.

### 17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

- 17.1 All sample preparation and analysis information will be documented on laboratory bench sheets, computer printouts, standard logbooks, etc. Raw data will be forwarded for reporting and for inclusion in the project files.

**STL - Pittsburgh**  
**Hardness Logsheet**

**Analyst:**

## Method

Date: \_\_\_\_\_ Start Time: \_\_\_\_\_

**Start Time:** \_\_\_\_\_

**Calcium** \_\_\_\_\_ **Good Fats** \_\_\_\_\_

Recd/Exp: \_\_\_\_\_

Calmagite  
Indicator Lot: \_\_\_\_\_ Recd/Exp: \_\_\_\_\_

Recd/Exp: \_\_\_\_\_

EDTA ID# \_\_\_\_\_ Normality: \_\_\_\_\_

**Normality:**

**Prep/Exp:** \_\_\_\_\_

True Value

LCS ID#: \_\_\_\_\_ /Range:

Remps:

**Prep/Exp:**

<u>LOT #</u>	<u>BATCH #</u>	<u>SDG #</u>
<p><b>Calculations:</b></p> $\text{mg CaCo}_3/\text{L} = \frac{[A \times N \times 50000]}{\text{mL Sample}} \quad \text{RPD: } \frac{[X1 - X2]}{\left(\frac{[X1 + X2]}{2}\right)} \times 100$ <p>A= mL EDTA titrant  N= Normality of titrant  X1=Orig Result X2=Dup</p>		

[illegible]

Date: \_\_\_\_\_

**GENERAL ELECTRIC COMPANY  
HUDSON RIVER BASELINE MONITORING PROGRAM (BMP)**

**Date:** May 11, 2006

**Organization Name:** Environmental Standards, Inc.

**Initiator's Name and Title:** Meg A. Michell – BMP QA Officer

**Problem Description:** Northeast Analytical, Inc. (NEA) discovered a long-term, systematic error in NEA's procedure for the preparation of water samples collected for the BMP prior to analysis of dissolved total organic carbon (DTOC). Once the whole water sample was centrifuged in order to separate the dissolved and particulate phases, the supernatant was being collected and sparged with nitrogen without first adding acid in order to remove inorganic carbon. The "DTOC" measurement that was being performed also measured inorganic carbon and was really a measurement of dissolved total carbon (DTC). NEA discovered the error while testing new TOC instrumentation this past winter, which allowed for automation of the acid addition and sparging procedure. The error was not discovered during either the laboratory auditing process or data validation process.

The DTOC analysis to be performed for the BMP is essentially a TOC analysis performed on the supernatant fraction of a water sample. NEA routinely receives non-BMP samples for TOC analysis that are acid preserved in the field and, as a result, only require nitrogen sparging to remove inorganic carbon during TOC analysis. In these cases, the addition of acid in the field serves a dual purpose – as a preservative to minimize microbial action and to acidify the sample prior to nitrogen sparging in order to remove inorganic carbon. As stated in the BMP QAPP (Table B-5), the samples collected for particulate organic carbon (POC) and DTOC analyses are not preserved in the field because the acid preservation would alter the dissolved/particulate relationship prior to separating the particulate and dissolved TOC fractions. The analyst systematically overlooked the necessity of the extra procedural step required for supernatant samples versus typical water samples (*i.e.*, to acidify the supernatant samples prior to nitrogen sparging) although the BMP QAPP SOP does state that acidification is necessary to remove inorganic carbon (BMP QAPP Appendix 19, Section 8.9.1). The BMP QAPP SOP does not specifically state that water samples collected for POC and DTOC analyses would not be preserved in the field and would always require acidification prior to nitrogen sparging.

NEA was acidifying the particulate fraction prior to sparging since solid samples collected for TOC analysis do not require field preservation with acid and therefore, all solid samples received by NEA require acidification prior to sparging for TOC analysis. As a result, the POC results are not impacted by this error.

Until January 15, 2006, the DTOC measurement has included the inorganic fraction and is a measurement of DTC (*i.e.*, is an overestimate of DTOC). DTC is an unnecessary parameter for the BMP, but DTOC is needed to assess partitioning of PCBs. From January 15, 2006 to April 11, 2006, NEA has performed paired analysis of DTOC and DTC. The attached table presents DTOC analyzed with and without addition of acid prior to the sparging step for several rounds of BMP data. A comparison of the results indicates that both inorganic carbon and organic carbon are present in the dissolved fraction and the differences are significant.

**Reported To:** Bob Gibson, GE; John Haggard, GE; John Connolly, QEA

---

**Corrective Action:** Until January 14, 2006, the DTOC results reported in the BMP database were actually DTC results. In addition, From January 15, 2006 to April 11, 2006, NEA was performing paired DTOC and DTC analyses but continuing to report the DTC results as DTC in the BMP database. As a result, the following changes have now been made in the BMP database.

- 1) The parameter name for all "Dissolved Total Organic Carbon" results in the BMP database have been changed to "Dissolved Total Carbon."
- 2) All available DTOC results have been added to the BMP database as DTOC.

NEA will also issue addendum data packages for each data set to correct reporting results as DTC or DTOC. The data summary reports (DSRs) that have been issued for the 2004 and 2005 field seasons and have already been sent to the Agency will be modified once comments have been received from the Agency.

Since April 12, 2006, NEA has been exclusively performing DTOC analyses and reporting the results as DTOC. The change in procedure will allow for a minimum of one year of DTOC data to be collected as part of the BMP. The data collected to date will be evaluated in order to evaluate if a relationship between DTOC and DTC can be determined.

The BMP QAPP SOP has been modified in order to state that samples that were not preserved with acid in the field (such as those collected for POC and DTOC analyses) require acidification prior to nitrogen sparging (attached Appendix 19 of BMP QAPP). In addition, a field will be added to NEA's TOC runlog for the notation of the addition of acid, including amount, type, concentration, and lot number of the acid. Laboratory auditors and data validation chemists will be instructed to verify that the acid addition takes place and/or is documented in the raw data.

**Approved By (USEPA RPM):** \_\_\_\_\_ **Date:** \_\_\_\_\_

**Reviewed and Implemented By:** David Blye (EnvStd)

cc: GE Program Manager: John Haggard; Bob Gibson  
QA Program Manager: David Blye (EnvStd)  
Other Distribution: John Connolly (QEA)  
Bob Wagner (NEA)

## TABLE

**DTC/DOC Comparison Table for Hudson River Baseline Monitoring Program**

<b>NEA Laboratory Record File (LRF) Number</b>	<b>NEA Batch Sample ID</b>	<b>NEA Sample ID</b>	<b>Client Sample ID</b>	<b>Date Collected</b>	<b>Dissolved Total Carbon (DTC)</b>	<b>Dissolved Total Organic Carbon (DOC)</b>	<b>Units</b>
0601130	01	AJ00738	RTN-060116-WF-C01	1/20/2006	11.3	3.13	mg/L
0601130	02	AJ00739	RTN-060116-ST-C01	1/19/2006	8.75	3.47	mg/L
0601130	03	AJ00740	RTN-060116-BF-FB01	1/19/2006	ND	ND	mg/L
0601130	04	AJ00741	RTN-060116-BF-C01	1/19/2006	6.79	3.70	mg/L
0601130	05	AJ00742	RTN-060116-BD-C01	1/19/2006	10.4	3.16	mg/L
0601130	06	AJ00743	HFL-060116-WF-C08	1/19/2006	11.6	3.18	mg/L
0601130	07	AJ00744	HFL-060116-WF-C07	1/19/2006	13.0	3.15	mg/L
0601130	08	AJ00745	HFL-060116-WF-C06	1/19/2006	13.5	3.67	mg/L
0601130	09	AJ00746	HFL-060116-WF-C05	1/18/2006	13.4	2.98	mg/L
0601130	10	AJ00747	HFL-060116-WF-C04	1/18/2006	13.8	3.01	mg/L
0601130	11	AJ00748	HFL-060116-WF-C03	1/18/2006	12.8	3.09	mg/L
0601130	12	AJ00749	HFL-060116-WF-C02	1/18/2006	12.5	3.02	mg/L
0601130	13	AJ00750	HFL-060116-WF-C01	1/15/2006	13.6	3.39	mg/L
0601162	01	AJ00972	RTN-060123-WF-C01	1/27/2006	12.4	2.97	mg/L
0601162	02	AJ00973	RTN-060123-ST-C01	1/26/2006	9.17	3.47	mg/L
0601162	03	AJ00974	RTN-060123-BF-FB01	1/26/2006	ND	ND	mg/L
0601162	04	AJ00975	RTN-060123-BF-C01	1/26/2006	7.12	4.09	mg/L
0601162	05	AJ00976	RTN-060123-BD-C01	1/27/2006	12.4	3.07	mg/L
0601174	01	AJ01118	RTN-060130-WF-C01	1/30/2006	12.2	3.37	mg/L
0601174	02	AJ01119	RTN-060130-ST-C01	1/30/2006	9.61	3.63	mg/L
0601174	03	AJ01120	RTN-060130-RI-C01	1/30/2006	7.09	ND	mg/L
0601174	04	AJ01121	RTN-060130-BF-FB01	1/31/2006	ND	ND	mg/L
0601174	05	AJ01122	RTN-060130-BF-C01	1/31/2006	7.29	4.33	mg/L
0601174	06	AJ01123	RTN-060130-BD-C01	1/30/2006	11.6	4.25	mg/L
0602031	01	AJ01310	RTN-060206-WF-C01	2/7/2006	10.3	2.77	mg/L
0602031	02	AJ01311	RTN-060206-ST-C01	2/7/2006	7.24	3.33	mg/L
0602031	03	AJ01312	RTN-060206-RI-C01	2/9/2006	6.60	3.97	mg/L
0602031	04	AJ01313	RTN-060206-BF-FB01	2/8/2006	ND	ND	mg/L
0602031	05	AJ01314	RTN-060206-BF-C01	2/8/2006	6.27	3.81	mg/L
0602031	06	AJ01315	RTN-060206-BD-C01	2/7/2006	10.2	2.78	mg/L
0602031	07	AJ01316	HFL-060206-WF-C02	2/8/2006	8.92	2.86	mg/L
0602031	08	AJ01317	HFL-060206-WF-C01	2/7/2006	9.96	2.84	mg/L
0602076	01	AJ01499	RTN-060220-WF-C01	2/21/2006	10.3	3.36	mg/L
0602076	02	AJ01500	RTN-060220-ST-C01	2/21/2006	8.37	4.35	mg/L
0602076	03	AJ01501	RTN-060220-RI-C01	2/22/2006	7.16	3.48	mg/L
0602076	04	AJ01502	RTN-060220-BF-FB01	2/22/2006	ND	ND	mg/L
0602076	05	AJ01503	RTN-060220-BF-C01	2/22/2006	6.04	3.71	mg/L
0602076	06	AJ01504	RTN-060220-BD-C01	2/22/2006	6.61	4.07	mg/L
0603061	01	AJ02196	RTN-060306-WF-C01	3/9/2006	12.2	3.70	mg/L
0603061	02	AJ02197	RTN-060306-ST-C01	3/9/2006	10.2	3.98	mg/L

**DTC/DOC Comparison Table for Hudson River Baseline Monitoring Program**

<b>NEA Laboratory Record File (LRF) Number</b>	<b>NEA Batch Sample ID</b>	<b>NEA Sample ID</b>	<b>Client Sample ID</b>	<b>Date Collected</b>	<b>Dissolved Total Carbon (DTC)</b>	<b>Dissolved Total Organic Carbon (DOC)</b>	<b>Units</b>
0603061	03	AJ02198	RTN-060306-RI-C01	3/9/2006	8.69	4.79	mg/L
0603061	04	AJ02199	RTN-060306-BF-FB01	3/10/2006	2.24	ND	mg/L
0603061	05	AJ02200	RTN-060306-BF-C01	3/10/2006	8.03	4.40	mg/L
0603061	06	AJ02201	RTN-060306-BD-C01	3/9/2006	9.63	4.73	mg/L
0603108	01	AJ02774	RTN-060313-WF-C01	3/15/2006	13.4	3.64	mg/L
0603108	02	AJ02775	RTN-060313-ST-C01	3/16/2006	9.42	3.80	mg/L
0603108	03	AJ02776	RTN-060313-RI-FB01	3/16/2006	2.89	ND	mg/L
0603108	04	AJ02777	RTN-060313-RI-C01	3/16/2006	8.38	3.49	mg/L
0603108	05	AJ02778	RTN-060313-BF-C01	3/17/2006	8.58	3.35	mg/L
0603108	06	AJ02779	RTN-060313-BD-C01	3/15/2006	13.4	3.96	mg/L
0603108	07	AJ02780	HFL-060313-WF-C01	3/14/2006	15.3	3.21	mg/L
0603167	01	AJ03393	RTN-060319-WF-C01	3/20/2006	9.93	3.92	mg/L
0603167	02	AJ03394	RTN-060319-ST-FB01	3/23/2006	ND	ND	mg/L
0603167	03	AJ03395	RTN-060319-ST-C01	3/23/2006	7.78	3.61	mg/L
0603167	04	AJ03396	RTN-060319-RI-C01	3/23/2006	7.25	4.42	mg/L
0603167	05	AJ03397	RTN-060319-BF-C01	3/24/2006	6.95	3.74	mg/L
0603167	06	AJ03398	RTN-060319-BD-C01	3/24/2006	7.01	3.93	mg/L
0603199	01	AJ03641	RTN-060327-WF-C01	3/29/2006	10.5	3.25	mg/L
0603199	02	AJ03642	RTN-060327-ST-C01	3/29/2006	7.54	3.38	mg/L
0603199	04	AJ03644	RTN-060327-RI-C01	3/29/2006	7.05	3.82	mg/L
0603199	05	AJ03645	RTN-060327-MR-C01	3/31/2006	21.0	2.15	mg/L
0603199	06	AJ03646	RTN-060327-BF-FB01	3/31/2006	ND	ND	mg/L
0603199	07	AJ03647	RTN-060327-BF-C01	3/31/2006	7.46	3.87	mg/L
0603199	08	AJ03648	RTN-060327-BD-C01	3/31/2006	20.5	2.25	mg/L
0604020	01	AJ03827	RTN-060403-WF-FB01	4/4/2006	ND	ND	mg/L
0604020	02	AJ03828	RTN-060403-WF-C01	4/4/2006	11.7	4.37	mg/L
0604020	03	AJ03829	RTN-060403-TI-C01	4/6/2006	7.20	3.43	mg/L
0604020	04	AJ03830	RTN-060403-ST-C01	4/4/2006	10.3	3.33	mg/L
0604020	05	AJ03831	RTN-060403-RI-C01	4/6/2006	6.36	3.53	mg/L
0604020	06	AJ03832	RTN-060403-BF-C01	4/6/2006	7.31	3.14	mg/L
0604020	07	AJ03833	RTN-060403-BD-C01	4/6/2006	7.70	3.40	mg/L
0604038	01	AJ03919	RTN-060410-WF-C01	4/10/2006	11.3	2.98	mg/L
0604038	02	AJ03920	RTN-060410-TI-C01	4/11/2006	7.62	4.53	mg/L
0604038	03	AJ03921	RTN-060410-ST-C01	4/11/2006	9.52	3.72	mg/L
0604038	04	AJ03922	RTN-060410-RI-FB01	4/10/2006	ND	ND	mg/L
0604038	05	AJ03923	RTN-060410-RI-C01	4/10/2006	7.37	3.65	mg/L
0604038	06	AJ03924	RTN-060410-BF-C01	4/11/2006	7.43	3.79	mg/L
0604038	07	AJ03925	RTN-060410-BD-C01	4/11/2006	8.65	3.41	mg/L

## **APPENDIX 19**

**STANDARD OPERATING PROCEDURE**

**NORTHEAST ANALYTICAL, INC.**

**NE128\_04.SOP**

**REVISION NUMBER: 04**

**STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF  
TOTAL ORGANIC CARBON (TOC) IN SOLIDS AND WATER AND THE  
DETERMINATION OF PARTICULATE (POC) AND DISSOLVED TOTAL  
ORGANIC CARBON (DIOC) IN WATER**

**MAY 2, 2006**

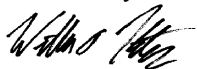
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## **STANDARD OPERATING PROCEDURE**

Author: Marsha Qian- Inorganics Manager

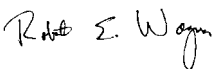
Northeast Analytical, Inc.  
Issuing section: Inorganics Department  
NE128\_04.SOP  
Date: 2-May-2006  
Revision Number: 4

Reviewed by:



William A. Kotas- QA Officer

Approved by:



Robert E. Wagner-Laboratory Director

### **1.0 TITLE**

Standard operating procedure for the determination of total and particulate organic carbon according to Tekmar-Dohrmann application note TOC-011.

### **2.0 PURPOSE**

The purpose of this SOP is to provide procedures for particulate organic carbon (POC) and total organic carbon (TOC).

### **3.0 SCOPE**

This method is applicable to waste water and ground water for POC and TOC, and sediments and filters for TOC.

### **4.0 COMMENTS**

Organic carbon is converted to carbon dioxide (CO<sub>2</sub>) by catalytic combustion or wet chemical oxidation. The CO<sub>2</sub> formed can be measured directly by an infrared detector. The amount of CO<sub>2</sub> is directly proportional to the concentration of carbonaceous material in the sample.

The fractions of total carbon (TC) are defined as:

- 1) inorganic carbon (IC)-the carbonate, bicarbonate, and dissolved CO<sub>2</sub>;
- 2) total organic carbon (TOC)-all carbon atoms covalently bonded in organic molecules;
- 3) dissolved organic carbon (DOC)-the fraction of TOC that passes through a 0.45-μm -pore-diameter filter,
- 4) particulate organic carbon (POC)-also referred to as non dissolved organic carbon, the fraction of TOC retained by a 0.45-μm filter.

IC interference can be eliminated by acidifying samples to pH 2 or less to convert IC species to CO<sub>2</sub>. Subsequently, purging the sample with a purified gas removes the CO<sub>2</sub>.

**Principle:** Depending upon the configuration, TOC can be measured by ultra-violet promoted persulfate oxidation or high-temperature combustion, followed by infrared detection.

- 1) TOC and POC in solid and sludge can be measured by utilizing the combustion-infrared method. The sample is homogenized and treated with acid and then heated to remove IC. The treated sample is placed into a heated reaction chamber packed with an oxidative catalyst such as cobalt oxide. The organic carbon is oxidized to CO<sub>2</sub> and H<sub>2</sub>O. The sludge and sediment sampler combusts samples at 800°C in an oxygen atmosphere so that solids as well as liquids can be analyzed.  
The sampler consists of a magnetically coupled boat inlet system which delivers the sample to the high temperature furnace. Two ports are provided for sample introduction, a septum port for liquid injections, and a flip-top port for solid samples. The CO<sub>2</sub> from the oxidation of organic carbon is transported in the carrier-gas stream and is measured by means of a nondispersive infrared analyzer (NDIR).
- 2) TOC in aqueous samples can be measured by UV promoted persulfate infrared method. External sparging is used to remove inorganic carbon. The acidified persulfate reagent is continuously pumped from the external reservoir to the injection port and then into the bottom of the UV reactor. The reactor is a constant volume design; the excess liquid is pumped to waste from the drain port. The reactor liquid is continuously sparged and this sparge/carrier gas flows out at the top of the reactor to the NDIR. When a sample containing combined carbon is injected, it is carried into the reactor by the reagent flow. The oxidation of organics occurs rapidly, and the resultant carbon dioxide is sparged from the liquid and carried to the NDIR.

The detection limit for samples is dependent on the amount of sample analyzed.

Note: If the determination of TOC, TC and IC is required for a water sample, an unfixed portion of the sample must be supplied and analyzed for TC. The inorganic carbon fraction of the sample is removed from an aliquot of the preserved sample which is then analyzed for TOC. The IC fraction of the sample is determined by taking the difference between the TOC and TC values.

**Sampling and storage:** The holding time for analyzing soil samples for TOC is 14 days from the date that the samples are collected. Samples are to be stored at 4°C until the time of analysis.

The holding time for analyzing water samples for TOC is 28 days from the date that the sample was collected. Collect samples in 40 ml VOA vials with silicone rubber-backed TFE septa with open ring caps. Preserve the samples with 1+1 H<sub>2</sub>SO<sub>4</sub> or 1+1 H<sub>3</sub>PO<sub>4</sub>. Samples are to be stored at 4°C until the time of analysis.

The holding time for analyzing water samples for POC is 14 days from the date that the sample was collected. Collect samples in one liter containers with Polyseal caps. Do not add any preservative to the bottles or samples. Samples are to be stored at 4°C until the time of analysis.

## 5.0 SAFETY

- 5.1 Safety glasses and disposable gloves must be worn when handling chemicals and samples.
- 5.2 Personnel should familiarize themselves with the necessary safety precautions by reading MSDS information covering any chemicals used to perform SOP.
- 5.3 Ultra-violet radiation can cause damage to the eyes. Do not open the door to the UV persulfate module without turning the lamp off.

## 6.0 REQUIREMENTS

- 6.1 **Method detection limit study.**

- 6.1.1 Seven MDLs samples (spike seven aliquots of laboratory water with the TOC standard) should be determined annually at a concentration of two to three times the estimated instrument detection limit for the analytes of interest.  
Analyze the samples according to the procedures set forth in this document. Calculate the MDL by multiplying the standard deviation of seven MDL measurements by 3.14. For the MDL to be valid, it must be greater than 1/10 the amount spiked but not greater than the amount spiked.
- 6.1.2 Knowledge on the operation and maintenance of the Dohrmann DC-80 series IR-I NDIR detector, UV-persulfate Reaction and sludge/sediment sampler modules.
- 6.1.3 Trainees are required to read the Instrument manual and take notes on subject matter not covered in SOP. Information about maintenance and replacement on specific parts not covered in SOP should be recorded on the "Notes" page of the SOP for future reference.

## **7.0 EQUIPMENT**

### **7.1 Equipment.**

- 7.1.1 Dohrmann IR-I NDIR detector module. Located in the main laboratory.
- 7.1.2 Dohrmann sludge/sediment sampler. Dohrmann (p/n 832-222). Located in the main laboratory.
- 7.1.3 250 and 1000 µL Rainin autopipets. Rainin (p/n EP-250 and EP-1000).
- 7.1.4 250 and 1000 µL pipet tips. Rainin (p/n RT-96 and RT-200).
- 7.1.5 1-5 ml Finn digital pipette with pipet tips. Baxter (p/n P5055-14).
- 7.1.6 Quartz boats. Dohrmann (p/n 899-624). Located in the main laboratory.
- 7.1.7 Quartz wool. Dohrmann (p/n 511-735). Located in the main laboratory.
- 7.1.8 GC oven. Set at 75 °C. Located in the main laboratory.
- 7.1.9 Propane tank with torch assembly. Located in the main laboratory.
- 7.1.10 Tweezers and steel spatula. Located in the main laboratory.
- 7.1.11 Analytical balance. Located in the main laboratory.
- 7.1.12 Centrifuge. Located in the main laboratory.
- 7.1.13 40 ml VOA vials. Located in the bottle storage room.
- 7.1.14 50, 100 and 250 µl syringe. Located in the main laboratory.
- 7.1.15 High purity oxygen tank with regulator. Located in the main laboratory.
- 7.1.16 Aluminum weighing boats. Located in the main laboratory.
- 7.1.17 Gray septum. Dohrmann (p/n 517-807). Located in the main laboratory.

- 7.1.18 Pasteur Pipets. Located in all laboratories.
- 7.1.19 UV-Persulfate Reaction Module. Located in the main laboratory.
- 7.1.20 Blue injection septum. Dohrmann (p/n 517-811). Located in the main laboratory.
- 7.1.21 Teflon sleeve reactor, taper joint. Dohrmann (p/n 070-627). Located in the main laboratory.
- 7.1.22 Lamp, Ultra-violet. Dohrmann (p/n 512-092). Located in the main laboratory.
- 7.1.23 Peristaltic pump tubing.
  - a) PVC Black/Black (p/n 899-641).
  - b) PVC Green/Green (p/n 899-645).
  - c) Viton A Purple/Purple (p/n 899-651).
- 7.1.24 High purity nitrogen tank with regulator. Attach plastic tubing to the regulator. Located in the main laboratory.
- 7.1.25 20-mesh tin. Dohrmann (p/n 511-876). Located in the main laboratory and used for tin/copper scrubber.
- 7.1.26 Copper. Dohrmann (p/n 511-895). Located in the main laboratory and used for tin/copper scrubber.
- 7.1.27 Pyrex wool. Dohrmann (p/n 511-895). Located in the main laboratory and used for tin/copper scrubber.

## **7.2 Reagents.**

- 7.2.1 Laboratory grade water. Located in the cooler room.
- 7.2.2 ~2500 mg/L TOC stock standard. Mallinkrodt (p/n 6704-1). Dry potassium hydrogen phthalate crystals (primary standard grade) in 104 °C oven for 2 hours and weigh out approximately 2.65675 grams. Record the weight in the Inorganic standard logbook and dissolve in approximately 400 ml of laboratory grade water, add 2 ml of phosphoric acid and bring to a final volume of 500 ml. Calculate the exact concentration of the solution:  

$$(\text{weight of potassium hydrogen phthalate}) \times 941 = \text{TOC stock standard } \{\text{mg/L}\}$$
- 7.2.3 TOCS and POC calibration standards:  
 Prepare 4 calibration standards of different concentrations ranging from ~120.7 - ~1207 mg/L. Record the date and information related to the preparation of the calibration standards in the Inorganic standard logbook.
- 7.2.4 TOC in water calibration standards (low level):  
 Prepare 5 calibration standards of different concentrations ranging from ~1.2 - ~24.10 mg/L. Record the date and information related to the preparation of the calibration standards in the Inorganic standard logbook.
- 7.2.5 7.9N (1+1) nitric acid. Dilute 50 ml of concentrated nitric acid to a final volume of 100 ml. Located in the Inorganics laboratory.
- 7.2.6 ICV/CCV: TOCS and POC 1000 mg/L TOC control. Ricca (p/n 1847-16). Located in the Inorganics laboratory.

- 7.2.7 ICV/CCV: TOC in water 10 mg/L TOC control. Ricca (p/n 1847-16). Dilute 1000 mg/L ICV/CCV standard 100x. Located in the Inorganics laboratory.
- 7.2.8 Concentrated phosphoric acid ( $\text{H}_3\text{PO}_4$ ). J.T. Baker. 'Baker analyzed', (Baxter p/n 0260-01\*BC). Located in the Inorganics laboratory.
- 7.2.9 2.0 % potassium persulfate. Add approximately 700 ml of laboratory water to a one liter volumetric flask and add 20 grams of  $\text{K}_2\text{S}_2\text{O}_8$  (J.T. Baker 'Baker Instra-analyzed' (Baxter p/n 3239-01\*BC)) to the flask with a stir-bar. Add 1 ml of concentrated phosphoric acid to the flask and stir until the  $\text{K}_2\text{S}_2\text{O}_8$  has dissolved. Remove the stir-bar and bring to volume.
- 7.3 Glassware and apparatus.**
- 7.3.1 10, 25, 50, 100 ml Class A volumetric flasks. Located in the Inorganics laboratory.
- 7.3.2 100 ml graduated cylinder. Located in the Inorganics laboratory.
- 7.3.3 Rinse bottle. Filled with laboratory grade water. Located in the Inorganics laboratory.
- 7.3.4 TOC logbook. Located next to TOC instrument.

## **8.0 PROCEDURE**

### **8.1 Operation and maintenance of the Dohrmann IR-I NDIR detector module.**

- 8.1.1 Refer to the instrument manual for specific instructions and part numbers for all components.
- 8.1.2 To prepare the tin/copper scrubber, fit one end of the Pyrex scrubber tube with a cored gray septum. Insert a tuft of Pyrex wool and then about 2 inches of tin in the other end. Secure the tin with another tuft of Pyrex wool. Then, fill the remaining half of the scrubber tube with an equal amount of copper. Secure the copper with a third tuft of Pyrex wool. Insert a cored gray septum. Inspect the tin/copper scrubber and change the contents of the tube when one-half of the tin is discolored.
- 8.1.3 The detector must be on for several hours in order to achieve equilibrium. It is recommended that the detector is turned on the day before the analysis is to be performed. Power up the detector and the main unit.
- 8.1.4 Verify that the printer has sufficient amount of paper before starting the analysis. Reset the printer so that the number "1" will be printed for the first analysis performed for that day.
- 8.1.5 Select the "TOC" and the "DET" positions. For the detector, select position "3" for high concentrations, "2" for medium concentrations, and "1" for low concentrations of TOC.
- 8.1.6 The module will not light the green "ready" light if the baseline is above 0.05. Adjust the "zero" control until the baseline is less than 0.02. The "CALIB" light must be off during analysis.

### **8.2 Operation and maintenance of the Dohrmann sludge/sediment sampler.**

- 8.2.1 Refer to the instrument manual for specific instructions and part numbers for all components.
- 8.2.2 A portion of sample is weighed into a quartz boat where it is acidified and dried. The boat is placed in the boat carriage of the sampler and it is moved into the combustion chamber. Gas from the combustion tube flows into the flask to the right where it passes through acidified water.

The gas travels to the flask to the left where excess water is removed before traveling to the detector module. The gas passes through the tin and copper scrubber and into the detector.

- 8.2.3 Before turning on the solid sampler, carefully examine individual components for sign of wear. Adjust the flow of oxygen to 30 psi. The level of acidified water in the right flask must be above the fritted sparging finger. A vigorous flow of gas emitting from the sparging finger should be easily observed, if not, check the gas lines and connections for leaks. The water collection flask should be emptied on a daily basis.
- 8.2.4 Turn on the furnace unit. When using the module for the first time or after a long period of inactivity, the furnace should be monitored with a voltmeter to verify that the temperature is at 800°C. Place the black (ground) probe in the "com" port. Place the red (positive) probe in the "monitor", set the voltmeter to "volts". The voltage reading should read "0.80", if not, place the red probe in the "adj" port. The voltage reading should read "0.80", if not, adjust the voltage by turning the set screw until the correct voltage is achieved.
- 8.2.5 If the gray septum (p/n 517-807) at either end of the combustion tube have corroded and require replacement, the furnace must be turned off before replacing the septum.

### **8.3 Calibration of Dohrmann sludge/sediment sampler and IR-I NDIR detector module.**

- 8.3.1 Determine the approximate concentration of the samples by analyzing one sample in each of the detector modes. Select the mode where the sample area readout is closest to the middle of the scale.
- 8.3.2 A new calibration curve must be generated if either the ICV or CCV (see **8.11 Quality Control**) are unacceptable. The calibration curve is based on 'µg of carbon' versus 'area'. Different volumes of the stock standard are injected onto a quartz boat that is lined with quartz wool. The calibration standards require duplicate injections.
- 8.3.3 A fresh tuft of quartz wool must be inserted into the boat before calibrating the instrument. The boat is placed inside the sediment sampler module. Hook the loop of the boat with the end of the magnetic boat carriage.
- 8.3.4 Remove contaminants from the boat by placing it in the furnace until the baseline has started to decrease. Pull the boat out of the furnace.
- 8.3.5 After the boat has cooled (approximately 30 seconds), place the boat underneath the injection port. Remove septum and inject calibration standard onto the boat. Replace septum.
- 8.3.6 After the baseline has stabilized, place the boat in the furnace. Press the "Start" button. After the signal has started to decrease, pull the boat out of the furnace.
- 8.3.7 Repeat injection of the standard until consecutive measurements are obtained that are reproducible to within  $\pm 10\%$ .
- 8.3.8 Repeat **8.3.5-7** for the remaining calibration standards.
- 8.3.9 The calibration and continuing check blank consists of 50 ml of laboratory water and one ml of 1+1 nitric acid. Inject 70 µl of the blank solution for the calibration and continuing check blanks.
- 8.3.10 For TOC solids and POC, inject 70 µl of each calibration standard and the stock standard. If the needle in the IR meter goes past '95' or if the red error light has lit after injecting the stock standard, inject a smaller volume of the standard. Every standard must be within the scale of the detector.

- 8.3.11 Enter the injection number, standard label, date analyzed, injection volume, and the area printed by the printer in the TOC logbook. See the **Glossary** for information about the correlation coefficient.

#### **8.4 Preparation of solid samples.**

- 8.4.1 Between 1.0 and 20 mg of material can be placed in a boat depending on the percent of carbon in the sample. Solid samples are analyzed in duplicate.
- 8.4.2 The concentration of the samples must be within the range of the calibration curve. If the sample concentration of the sample is outside the range of the calibration curve, repeat the analysis of the sample. If the  $\mu\text{g}$  of carbon of the sample was too low, use more sample up to 20 mg. If the sample concentration was too high, use less sample down to 1.0 mg.
- 8.4.3 Place each boat in a numbered aluminum weigh boat.
- 8.4.4 Homogenize a portion of the sample.
- 8.4.5 Place one boat on the analytical balance and tare the balance. Transfer an aliquot of the sample to the boat and record the NEA #, weight and the boat number in the TOC logbook. Place the boat in the numbered aluminum weigh boat.
- 8.4.6 Repeat **8.4.5** for the replicate sample analysis.
- 8.4.7 Add 2 to 3 drops of 1+1 nitric acid to each sample. Turn off the GC oven. Place the aluminum weigh boats in the GC oven. Place a 60 ml beaker over each quartz boat. Turn on the GC oven. Remove samples when dried (minimum of 10 minutes).
- 8.4.8 Place the boat in the raceway. After the baseline has stabilized, place the boat in the furnace and press the 'Start' button.
- 8.4.9 Copy the TOC area from the printer into the TOC logbook.
- 8.4.10 After each sample analysis, scrape any remaining material from the boat and place the boat in the flame of the propane torch to remove any contaminants.
- 8.4.11 Repeat **8.4.3-9** for the remaining samples.

#### **8.5 Percent total solids determination**

- 8.5.1 Determine the percent total solids for each sample as described in NE090.

#### **8.6 The determination of Particulate organic carbon (POC) in water.**

- 8.6.1 The purpose of this procedure is to separate the non dissolved TC compounds from the dissolved TC compounds by centrifuging the water sample. The IC fraction of the sample is removed by the addition of 1+1 nitric acid to the particulate matter. Note: If DTOC (Dissolved Total Organic Carbon) analysis is also required a portion of the supernatant (upper layer) is removed for subsequent analysis via persulfate oxidation/aqueous injection method described in section 8.9.

- 8.6.2 Shake the sample bottle and measure a maximum of 80 ml aliquot of the sample with a graduated cylinder. Pour the sample into two labeled volatile (VOA) vials. Verify that sample levels in each vial are equal to each other.
- 8.6.3 Centrifuge the VOA vials at a setting of '7' for 5 minutes
- 8.6.4 Remove all of the water (supernatant) from each vial until approximately 10 ml remain in each vial. If the sample requires DTOC determination, acidify the removed supernatant with 1:1 H<sub>3</sub>PO<sub>4</sub> and store in a clean VOA vial for subsequent analysis as described in section 8.9 below:
- 8.6.5 Transfer all the material (water and particulates) from the two vials to one vial.
- 8.6.6 Centrifuge the vial with the water and particulates at a setting of '7' for 5 minutes.
- 8.6.7 Remove all the water from the vial. Set the 1000 µl Rainin pipet to 650 µl and transfer the particulates to a quartz boat.  
Note: If all the material from the VOA vial will not fit inside the boat, transfer a portion of the material from the vial to the boat and dry the boat and the material inside the GC oven. Repeat the process of transferring the sample from the vial to the boat and drying the material until all the sample extract has been transferred to the boat.
- 8.6.8 Place the boat in the numbered aluminum weigh boat. Record the NEA #, volume of sample centrifuged and the boat number into the TOC logbook.
- 8.6.9 Add 2 to 3 drops of 1+1 nitric acid to each sample. Turn off the GC oven.. Place the aluminum weigh boats in the GC oven. Place a 60 ml beaker over each quartz boat. Turn on the GC oven. Remove samples when dried (minimum of 10 minutes).
- 8.6.10 Follow the instructions in **8.3.6** for analyzing samples.
- 8.6.11 The concentration of the samples must be within the range of the calibration curve. If the sample concentration was too high, extract less than 80 ml of the sample.
- 8.7 Set up and maintenance of the UV-Persulfate reaction module.**
- 8.7.1 Refer to the instrument manual for specific instructions and part numbers for all components.
- 8.7.2 Connect the tubing from the oxygen tank to the 'Carrier in' port. Connect the tubing from the 'Carrier out' port to the 'In' port of the NDIR detector module.
- 8.7.3 For the UV lamp, a thin film of Teflon fabricated in a conical shape is placed over the taper joint. Any excess is trimmed back from the top and bottom ends of the joint with a razor blade.
- 8.7.4 The position of the lamp should be adjusted so that the reactor coils just clears the fritted glass gas dispenser. Carefully insert the cap and lamp assembly into the reactor and check the clearance to the fritted gas dispenser. The lamp is held together by two springs.
- 8.7.5 Install the lamp so that the carrier gas exit tube is pointing to the front. The reactor is held in place by a three prong grip utility clamp.

- 8.7.6 **Reactor Liquid Plumbing.** Connections are made between the ports on the inside of the right side of the module to the reactor and other ports inside the reactor with Teflon lines and red/white septums. The ports on the inside of the module are counted one through six starting with one near the top of the case.
- 8.7.7 Connect the Teflon line from port 1 to the waste drain port of the reactor (the top of the "U" tube on the right side of the reactor). The 1/16" line should be pushed down through the septum about one inch and later adjusted up or down in the side arm so that the liquid level is about 1/4" above the inlet of the recycle arm.
- 8.7.8 Connect the line from port 2 to the recycle arm of the reactor. This port is located near the upper-center and points upward. Plug the horizontal port of this side arm with a red/white septum without a hole.
- 8.7.9 Connect the free line from the injection port to the sample inlet port at the bottom-left of the reactor body. Insert the Teflon tubing almost all the way through the glass capillary section of the inlet port.
- 8.7.10 **Reactor Gas Plumbing and Liquid Trap Plumbing.**
- 8.7.11 Connect 1/8" Teflon line from port 4 to the gas inlet at base of reactor with a red/white septum.
- 8.7.12 Mount liquid trap "U" tube to the left of the reactor and run drain line to a beaker under the reactor.
- 8.7.13 Connect 1/8" Teflon line from outlet port of reactor cap to top inlet port of liquid trap with a red/white septum at reactor end and a gray perforated septum at the "U" tube end. Push line at the "U" tube inlet through septum hole until it is just below top of bulb.
- 8.7.14 Connect 1/8" Teflon line from permeation drier (top left of the interior of the module to the angled port of the "U" tube) with a gray perforated septum.
- 8.7.15 **Reaction Module Pump Tube Installation and Pump Adjustment.**
- 8.7.16 Release the pump tube pressure fingers by pressing on the upper part of the white plastic plate located toward the front of the pump assembly. This will release the pressure plate and allow the pressure fingers to rotate downward.
- 8.7.17 Install a green/green bridged tube at the inner most position. Install a black/black bridged tube in the second position. Install a black Viton purple/purple bridged tube in the third position.  
Note: The pump pressure plate and fingers should be left in their operating position overnight to insure that reagent does not siphon out of the reactor.
- 8.7.18 Raise all four pressure fingers and raise the pressure plate so that the screws press up on the fingers. Push up on the bottom of the pressure plate and push in on the bottom of the white plastic locking block until it locks the pressure plate in place.
- 8.7.19 Connect a piece of plastic tubing to the back end of the green/green tubing. Place the free end of the plastic tubing into a container of laboratory water. Turn on the pump. Slowly adjust the screw for the green/green tube inward until the water just starts to rise in the tube. Advance the screw one-half turn more.
- 8.7.20 Repeat the procedure for the black/black and purple/purple tubes.
- 8.7.21 **Reactor External Plumbing.** The pump tube inlets are to the rear, outlets to the front. Connections between the pump tubing and module tubing are made on the ports outside the module.

- 8.7.22 Connect the inlet of the green/green pump tube to port 1 with 1/8" Teflon line. Connect a 1/8" Teflon line to the outlet of the green/green pump tube and place the end of the line in a waste container on the bench top.
- 8.7.23 Connect a 1/16" Teflon line to the inlet of the black/black pump tube and place the end of the line in the 2.0%  $K_2S_2O_8$  solution.
- 8.7.24 Connect a 1/16" Teflon line between port 3 and the exit side (left) of the mixing tee. Connect a 1/16" Teflon line between the outlet of the black/black pump tube and the top of the mixing tee. Connect a 1/8" Teflon line between the outlet of the purple/purple pump tube and the mixing tee.
- 8.7.25 Connect a 1/8" Teflon line between port 2 and the outlet of the purple/purple pump tube.

## **8.8 Operation and calibration of the UV-persulfate and IR-I NDIR detector modules.**

- 8.8.1 The blue injection septum must be replaced after approximately 100 injections have been made. Replace the blue septum in the injection port before starting the flow of reagent through the module.
- 8.8.2 Because there is a low flow of reagent(s) to the reactor, gently remove the top of the reactor and pour 2.0%  $K_2S_2O_8$  so that the time required to fill the reactor with reagent is decreased. The reactor should be approximately 2/3 full. Connect the top of the reactor.
- 8.8.3 Place the pressure plate on the pump tubes. Turn on the pump and lamp by pressing the three white power buttons.  
Caution: Do not open the module door while lamp is on.
- 8.8.4 Turn on the NDIR detector. The level of reagent in the reactor recycle arm must be at the top of the arm before starting analysis. The baseline on the detector must also be stable before starting analysis.
- 8.8.5 Inject the calibration standards one at a time. Wait 15 seconds and Press the "Start" button. The instrument will 'beep' to indicate that the analysis is completed and is ready for the next injection. Repeat injection of the sample until consecutive measurements are obtained that are reproducible to within  $\pm 10\%$ .
- 8.8.6 For low level analysis, the amount of carbon inject into the instrument for the calibration standards should range from  $\sim 0.10$  to  $24 \mu g$ . This can be achieved by injecting different volumes of the calibration standards.
- 8.8.7 Enter the injection number, standard label, date analyzed, injection volume, and the area printed by the printer in the TOC logbook. See the **Glossary** for information about the correlation coefficient.
- 8.8.8 After the analysis is completed, flush the reactor system by placing the tubing for the 2.0 %  $K_2S_2O_8$  in a container of RO water and turn the pump on for one hour.

## **8.9 The determination of TOC and Dissolved TOC (DTOC) in water.**

- 8.9.1 Remove the IC fraction of the sample by acidifying (if the sample was not previously acidified) and sparging the preserved (acidified) sample with nitrogen gas for 10 minutes.
- 8.9.2 Transfer a portion of the sample to a VOA vial and place in a test tube rack. Attach a Pasteur pipet to plastic tubing that is connected to a nitrogen tank.

Add three drops of concentrated  $\text{H}_3\text{PO}_4$  to the sample and place the tip of the Pasteur pipet in the sample. Slowly turn on the gas flow to produce gentle bubbling inside the vial for 10 minutes. The sample is now ready for analysis.

- 8.9.3 Inject 0.100 ml of sample into the UV-persulfate module. Wait 15 seconds and Press the "Start" button. The instrument will 'beep' to indicate that the analysis is completed and is ready for the next injection. Repeat injection of the sample until consecutive measurements are obtained that are reproducible to within  $\pm 10\%$ .
- 8.9.4 The concentration of the samples must be within the range of the calibration curve. If the original concentration of the sample was too low, inject a larger volume of sample up to 0.25 ml. If the sample concentration was too high, inject a smaller volume down to 0.010 ml. If the sample concentration is still too high, dilute a portion of the unsparged sample and repeat **8.7.2** and re analyze the diluted sample.
- 8.9.5 Repeat **8.9.2-4** for the remaining samples.
- 8.9.6 Enter the injection number, standard label, date analyzed, injection volume, and the area printed by the printer in the TOC logbook.

#### **8.10 Sample calculations utilizing Lotus spreadsheets.**

- 8.10.1 After the instrument is calibrated, a Lotus spreadsheet is used to construct a calibration curve and the linear regression. Generate a spreadsheet each time that the instrument is calibrated for either water samples or solids.
- 8.10.2 Log into the network and access "Lotus 1-2-3". Recall a previous spreadsheet, see the following table for an example of the directories and examples of files saved on November 11, 1996.

Analyte (matrix)	Lotus directory	Example
TOC (solids)	S:\DATA\TOCS*.*	S:\DATA\TOCS\1118.WK6
TOC (water)	S:\DATA\TOC*.*	S:\DATA\TOC\1118.WK6
POC (water)	S:\DATA\POC*.*	S:\DATA\POC\1118.WK6

- 8.10.3 Enter the average area (subtract the average blank area) for the calibration standards in the box used for constructing the calibration curve. Update the linear regression. For the calibration curve, enter the date of analysis.
- Note:** Except for the lowest calibration standard, the percent recoveries for the calibration standards must be between 90 and 110%.

#### **8.11 Quality control (see attachment B for corrective actions)**

- 8.11.1 A calibration blank is required for each day of analysis. Check blanks are analyzed after every initial and continuing check standard. The concentration of the blank must be less than the MDL for that method.
- 8.11.3 **Sample duplicate:** A duplicate analysis is performed every 10 samples.  
 $\text{RPD} = \text{Abs. } \{(S1 - S2)/(S1 + S2)\} \times 200$
- 8.11.4 **Independent and continuing calibration verification standard (ICV) and (CCV):** A purchased TOC solution of known concentration is analyzed after each calibration curve is generated, after every 10 samples and at the beginning and end of the analysis.  
 The ICV/CCV is analyzed in replicate.

$\% \text{ recovery} = (\text{calculated value} / \text{certified value}) \times 100.$

8.11.5 For soil samples, if the sample analyses was off scale and the minimum sample weight of 1.0 mg was used, calculate the maximum concentration of TOC based on the  $\mu\text{g}$  of carbon of the highest calibration standard, average sample weight, and the percent total solids. Report the results as greater than the calculated maximum sample concentration, the detection limit and the standard deviation

8.11.6 **Laboratory fortified sample matrix.** Perform a spike on every 20<sup>th</sup> soil or water sample. For water samples, spike 10 ml of the sample with an aliquot of the ICV/CCV standard and proceed as in **8.9.2-4**. For soil samples, weigh the sample and proceed as in **8.4.1-10**. Place the sample and boat in the boat sampler and spike the sample through the injection port with the ICV/CCV standard. The final concentration of the spiked sample must be within the calibration curve.

$\% \text{ recovery} = \{(\text{spike sample conc.}) - (\text{sample conc.})\} / (\text{spike added}) \times 100$

## **8.12 Entry of data into LIMs.**

8.12.1 After the calibration curve has been completed, give the LIMs manager a copy of the Lotus spreadsheet for the calibration curve with the area for blank and area for the lowest standard used in the calibration curve.

8.12.2 Log into LIMS. Click "Win Results" or "Results" from LIMS toolbar. Select the appropriate samples by either typing in the sample ID's or selecting the Login Record File.

8.12.3 Choose the result entry template "TOCSOL", then click "OK". A result entry spreadsheet will then be created with the following columns: TOCBLANK, TOCSLOPE, \$TOCAREA, \$TOCWTWG, \$TOCFINL, %SOLIDS. To find out what should go into these QC data columns, right click on the column heading in gray at the top of the spreadsheet.

8.12.4 The data for samples should be entered into the columns as follows:

\$TOCBLANK = Calibration Blank Absorption

\$TOCSLOPE = Inverse Slope Absorption

\$TOCAREA = Area Counts for Sample

\$TOCWTWG = Sample weight in grams

%SOLIDS = % Total Solids for Sample (Enter as a percentage, not a decimal)

\$TOCFINL = Final result for TOC in Solids (Fills in automatically) along with the Average and %RSD

8.12.5 Once the field \$TOCFINL has been filled in by the computer, right click on that field and select "detailed edit" from the pull down menu. Confirm that the MDL and the date analyzed for the sample are correct. Proceed to the next sample.

8.12.6 Once the data has been entered for all samples, go the QC section of the spreadsheet. If batching was performed correctly there should be some of these fields displayed in white. Right click on the dark gray fields in that same row so that all appropriate QC tests have been added.

(For example, if the sample has a duplicate be sure all the raw data fields for the duplicate have been turned white.) Enter in all appropriate QA/QC data.

## **9.0 REFERENCES**

9.1 "Determination of Total Organic carbon in sediment," Lloyd Kahn, U.S.E.P.A. Region II, Edison NJ.

- 9.2 Application Note: TOC-011 "Analysis of sludges and solids for carbon," Tekmar-Dohrmann, Cincinnati, OH 10/95.
- 9.3 NYSDOH ELAP manual item #271, 4/15/94.
- 9.4 *Standard Methods for the Examination of Water and Wastes*, method #5310B, 17th edition. 1989.

## 10 ATTACHMENTS

- 10.1 **Attachment A:** Note pages for analyst.
- 10.2 **Attachment B:** Quality assurance and corrective action for problems associated with sample preparation and analysis.
- 10.3 Attachment C: Disposal of samples and waste.

# **ATTACHMENT A NOTES**

**ATTACHMENT A CONTINUED  
NOTES**

## ATTACHMENT B

### QUALITY ASSURANCE AND CORRECTIVE ACTIONS.

**Calibration curve:** If the correlation coefficient is  $< 0.997$  or if the recoveries for any of the calibration standards are not within 10% of the true value (except for the lowest standard), repeat injections of the outlying standards until curve is within acceptance criteria.

**Independent calibration verification (ICV/QCS):** Must be within 85 –115% of true value. If the Percent recovery is not within the limits specified, recalibrate the instrument and reanalyze all samples since the last compliant continuing calibration verification standard.

**Check standard (CCV/IPC):** Use ICV solution as mentioned previously. Must be within 85 –115% of true value. If the Percent recovery is not within the limits specified, recalibrate the instrument and reanalyze all samples since the last compliant continuing calibration verification standard.

**Preparation blank:** For TOC in water, prepare one blank consisting of laboratory grade water for each batch of samples sparged daily. Prepare blank as described in 8.91-3. If the average area of the blank is greater than the half the value of the lowest standard used to construct the calibration curve, prepare a new blank solution.

**Check blank (CCB):** For TOC in water, see **Preparation blank**. For TOC in solids, analyze 70 ul of laboratory grade water. Analyze the CCB solution after each ICV/CCV solution. If the average area of the blank is greater than the half the value of the lowest standard used to construct the calibration curve, determine the source of the problem, fix the problem and reanalyze all samples since the last compliant CCB.

**Laboratory control sample:** Not applicable

**Sample duplicate:** Prepare and analyze one sample duplicate for every 10<sup>th</sup> sample. For water samples, a control limit of 20% for RPD shall be used for original and duplicate sample values greater than or equal to 5x the RDL. A control of +/- the RDL shall be used if either the sample or its duplicate is less than 5x the RDL. For soil samples, refer to the latest control limit for duplicates. If the results for the sample and duplicate are unacceptable, a case narrative explaining why the RPD for a sample and its duplicate was outside the control limits must be written and approved by the quality assurance officer. A copy of the case narrative must be sent with the report to the client.

**Matrix spike:** Prepare and analyze one matrix spike for every 20<sup>th</sup> sample. **TOC:** For water and soil samples, refer to the latest control limit for matrix spikes. Spike with an aliquot of the ICV/CCV solution. If the results for the matrix spike is unacceptable, prepare and analyze another matrix spike. If the results for the matrix spike is still unacceptable, a case narrative explaining why the percent recovery for the matrix spike was outside the control limits must be written and approved by the quality assurance officer. A copy of the case narrative must be sent with the report to the client

**Serial dilution:** Not applicable

**Analytical spike:** Not applicable

**Method of standard additions:** Not applicable

**Overrange samples:** Dilute or redigest samples that are greater than the value of the highest standard used to prepare the calibration curve so that the results are within the calibration curve.

## **ATTACHMENT C: DISPOSAL OF SAMPLES AND WASTE**

1. Refer to SOP NE054 for procedures for disposing of laboratory waste.
2. Acidified aqueous samples and extracts that do not contain metals or organic compounds above 0.050 mg/L, can be neutralized to a pH above 4.0 before disposal.
3. All client sample containers must be defaced with a permanent marker before disposal.

## 11 GLOSSARY

- 11.1 Laboratory control.:** A standard of known concentration that is independent of the standards used for quantifying samples.
- 11.2 Continuing calibration standard (CCV):** Used to assure calibration accuracy during each analysis run. It must be run at a frequency of 10% during the run. It must also be analyzed at the beginning and the end of the run. Its concentration must be at or near the mid-range level of the calibration curve.
- 11.3 Correlation coefficient:** The correlation coefficient for the calibration curve must be greater than or equal to 0.997 according to NYSDOH requirements.

**APPENDIX C**  
**BMP PROJECT DATABASE**  
**(CD-ROM ATTACHED)**

**Hudson River Baseline Monitoring Program**  
USEPA Fish Database Export Dictionary

**EVENTS**

#	Attribute Name	Description	Data Type (Size)	Units	Notes
1	SAMPLING_EVENT_ID	Unique sampling event ID. Used to link fish EVENTS table with DESCRIPTION table.	Text(15)		Example: "ND1-040603-01"
2	STATION_ID	Sampling location abbreviation.	Text(5)		
3	STATION_NAME	Full location name.	Text(50)		
4	SAMPLE_COLLECT_METHOD	Indicates fish sample collection method.	Text(5)		NET: netting ES: electroshocking ANG: angling
5	EVENT_START_DATE_TIME	Date and time sampling event initiated.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
6	EVENT_END_DATE_TIME	Date and time sampling event completed.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
7	START_NORTHING	Northing coordinate of upstream end of sampling location (NY state plane east NAD83).	Text(20)	ft	
8	START_EASTING	Easting coordinate of upstream end of sampling location (NY state plane east NAD83).	Text(20)	ft	
9	END_NORTHING	Northing coordinate of downstream end of sampling location (NY state plane east NAD83).	Text(20)	ft	
10	END_EASTING	Easting coordinate of downstream end of sampling location (NY state plane east NAD83).	Text(20)	ft	
11	COORDINATE_UNIT	Unit of measurement for the northing and easting coordinates.	Text(15)		
12	WATER_TEMP	Temperature of water at beginning of sampling event.	Double	Degrees C	
13	TURBIDITY	Turbidity of water at beginning of sampling event.	Double	see Field 14	
14	TURBIDITY_UNITS	Unit of measurement for turbidity.	Text(5)		
15	CONDUCTIVITY	Conductivity of water at beginning of sampling event.	Double	see Field 16	
16	CONDUCTIVITY_UNITS	Unit of measurement for conductivity.	Text(5)		
17	WEATHER	Weather conditions during sampling event.	Text(100)		

**Hudson River Baseline Monitoring Program**  
USEPA Fish Database Export Dictionary

#	Attribute Name	Description	Data Type (Size)	Units	Notes
18	COMMENTS	General comments or field observations.	Text(255)		
19	SAMPLER_INITIALS	Initials of sampler.	Text(5)		

**Hudson River Baseline Monitoring Program**  
USEPA Fish Database Export Dictionary

**DESCRIPTION**

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
1	FIELD_SAMPLE_ID	Unique field sample ID. Used to link to Results tables.	Text(50)		Example: "RTN-040609-L1-C01"
2	SAMPLING_EVENT_ID	Event ID for fish sample. Used to link fish EVENTS table with DESCRIPTION table.	Text(15)		
3	SAMPLE_MATRIX_CODE	Code which distinguishes between different types of sample matrix.	Text(50)		"F"
4	SAMPLE_TYPE_CODE	Code which distinguishes between different types of samples.	Text(50)		"ENV"
5	SAMPLE_SOURCE	This field identifies where the sample came from.	Text(10)		"Field" or "Lab"
6	SAMPLE_DATE_TIME	Date and time sample was collected.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
7	CHAIN_OF_CUSTODY	Chain of custody identifier.	Text(50)		Example: "COC040603-A01-01"
8	SAMPLE_ARCHIVED	Indicates if a sample was archived.	Text(50)		"Yes" or "No"
9	ARCHIVE_ONLY	Indicates if a sample was archived only.	Text(50)		"Yes" or "No"
10	EPA_SPLIT	Indicates if the sample was chosen as a split by the USEPA.	Text(50)		"Yes" or "No"
11	COMPOSITE_YN	Indicates if sample is a composite.	Text(50)		"Yes" or "No"
12	NUM_IN_COMPOSITE	If composite sample, indicates number of fish in composite.	Long Integer		
13	SPECIES_CODE	NYSDEC abbreviations for species of fish sample.	Text(20)		
14	TOTAL_LENGTH	Total length of fish sample for individuals (nearest mm).	Long Integer	mm	
15	LENGTH_UNIT	Unit of measurement for length.	Text(5)		
16	WEIGHT	Total weight of fish sample for individuals (nearest 0.1 grams).	Double	g	
17	WEIGHT_UNIT	Unit of measurement for weight.	Text(5)		
18	SEX	Sex of fish sample.	Text(5)		M: male F: female U: unknown
19	AGE	Age of individual fish.	Long Integer		
20	SAMPLE_PREP	Indicates type of sample collected.	Text(20)		"fillet" or "whole body"
21	GENERAL_DESCRIPTION	General comments or field observations at time of sample collection.	Text(255)		
22	CPUE_ID	Corresponding ID from "catch per unit effort" sampling.	Text(30)		

**Hudson River Baseline Monitoring Program**  
USEPA Fish Database Export Dictionary

**RESULTS PCBs and RESULTS NONPCBs**

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
1	FIELD_SAMPLE_ID	Unique field sample ID.	Text(50)		Example: "RTN-040609-L1-C01"
2	LAB_SAMPLE_ID	Laboratory sample identifier.	Text(255)		
3	SAMPLE_TYPE_CODE	Code which distinguishes between different types of sample.	Text(25)		ENV: environmental sample MB: method blank LCS: laboratory control sample MS: matrix spike MSD: matrix spike duplicate
4	SAMPLE_MATRIX_CODE	Code which distinguishes between different types of sample matrix.	Text(25)		"F"
5	SAMPLE_SOURCE	This field identifies where the sample came from.	Text(10)		"Lab" or "Field"
6	SAMPLE_COMMENT	Sample comments as necessary.	Text(255)		
7	LAB_ANALYTICAL_METHOD	Laboratory analytical method name or description.	Text(50)		
8	ANALYSIS_DATE_TIME	Date of sample analysis.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
9	TOTAL_OR DISSOLVED	"T" for total (metal) concentration, "D" for dissolved or filtered (metal) concentration, or "N" for organic (or other) constituents for which neither "total" nor "dissolved" is applicable.	Text(1)		"T", "D", or "N"
10	COLUMN_NUMBER	"1C" for first column analyses, "2C" for second column analyses, or "NA" for analyses for which neither "1C" nor "2C" is applicable.	Text(5)		"1C", "2C", or "NA"
11	TEST_TYPE	Type of test.	Text(20)		"initial", "reextract", and "reanalysis"
12	CAS_RN	Chemical Abstracts Registry Number for the parameter if available.	Text(20)		
13	PARAMETER	Chemical name.	Text(60)		
14	RESULT_VALUE	Analytical result.	Double		blank for non-detects
15	RESULT_ERROR_DELTA	Error range applicable to the result value.	Double		

**Hudson River Baseline Monitoring Program**  
USEPA Fish Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
16	RESULT_TYPE_CODE	"TRG" for a target or regular result, and "SUR" for surrogates.	Text(10)		"TRG" or "SUR"
17	REPORTABLE_RESULT	"Yes" for results which are considered to be reportable, or "No" for non-reportable results.	Text(10)		"Yes" or "No"
18	DETECT_FLAG	"Y" for detected analytes or "N" for non-detects.	Text(3)		"Y" or "N"
19	QC_LEVEL	Status of data quality review.	Text(50)		"Verified" or "Validated"
20	RESULT_QUALIFIERS	Qualifiers assigned to samples during data verification /validation.	Text(50)		
21	ORGANIC_YN	"Y" for organic constituents or "N" for inorganic constituents.	Text(3)		"Y" or "N"
22	MDL	Method detection limit.	Double		
23	RL	Detection limit that reflects conditions such as dilution factors and moisture content.	Double		
24	QL	Concentration level above which results can be quantified with confidence.	Double		
25	RESULT_UNIT	Units of measurement for the result.	Text(15)		
26	DETECTION_LIMIT_UNIT	Units of measurement for the detection limit(s).	Text(15)		
27	RESULT_COMMENT	Result specific comments.	Text(255)		
28	QC_ORIGINAL_CONC	The concentration of the analyte in the original (unspiked) sample.	Double		
29	QC_SPIKE_ADDED	The concentration of the analyte added to the original sample.	Double		
30	QC_SPIKE_MEASURED	The measured concentration of the analyte.	Double		
31	QC_SPIKE_RECOVERY	The percent recovery calculated.	Double		
32	QC_DUP_ORIGINAL_CONC	The concentration of the analyte in the original sample.	Double		
33	QC_DUP_SPIKE_ADDED	The concentration of the analyte added to the original sample.	Double		
34	QC_DUP_SPIKE_MEASURED	The measured concentration of the analyte	Double		

**Hudson River Baseline Monitoring Program**  
USEPA Fish Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
		in the duplicate (for background corrected matrix spike duplicates).			
35	QC_DUP_SPIKE_RECOVERY	The duplicate percent recovery calculated.	Double		
36	QC_RPD	The relative percent difference calculated.	Double		
37	QC_SPIKE_LCL	Lower control limit for spike recovery.	Double		
38	QC_SPIKE_UCL	Upper control limit for spike recovery.	Double		
39	QC_RPD_CL	Relative percent difference control limit.	Double		
40	QC_SPIKE_STATUS	Indicates whether the spike recovery was within control limits. The "*" character indicates failure; otherwise blank.	Text(20)		
41	QC_DUP_SPIKE_STATUS	Indicates whether the duplicate spike recovery was within control limits. The "*" character indicates failure; otherwise blank.	Text(20)		
42	QC_RPD_STATUS	Indicates whether the relative percent difference was within control limits. The "*" character indicates failure; otherwise blank.	Text(20)		
43	LAB_MATRIX_CODE	Code which distinguishes between different types of lab sample matrix.	Text(10)		"F"
44	ANALYSIS_LOCATION	"FI" for field instrument or probe, "FL" for mobile field laboratory analysis, or "LB" for fixed-based laboratory analysis.	Text(2)		"FI", "FL", or "LB"
45	BASIS	"Wet" for wet-weight basis reporting, "Dry" for dry-weight basis reporting, or "NA" for tests for which this distinction is not applicable.	Text(10)		"WET", "DRY", or "NA"
46	DILUTION_FACTOR	Effective test dilution factor.	Double		
47	PRFP_METHOD	Laboratory sample preparation method name or description.	Text(50)		Yes
48	PREP_DATE_TIME	Date and time of sample preparation military format.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
49	LAB_NAME_CODE	Unique identifier of the laboratory.	Text(15)		

**Hudson River Baseline Monitoring Program**  
USEPA Fish Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
50	DATA_PACKAGE_LEVEL	Data package level.	Text(10)		"A", "B", or "AB"
51	PERCENT_MOISTURE	Percent moisture of the sample portion used in this test.	Double		
52	SUBSAMPLE_AMOUNT	Amount of original sample used in sample preparation.	Double		
53	SUBSAMPLE_AMOUNT_UNIT	Unit of measurement for subsample amount.	Text(15)		
54	SAMPLE_DELIVERY_GROUP	Sample delivery group.	Text(20)		
55	TEST_COMMENT	Comments about the test as necessary.	Text(255)		
56	FINAL_VOLUME	The final amount/volume of the sample, extract, or digestate after sample preparation.	Double		
57	FINAL_VOLUME_UNIT	The unit of measure that corresponds to the final volume.	Text(15)		
58	PREP_BATCH_ID	ID for unique prep batch.	Text(15)		
59	ANALYSIS_BATCH_ID	ID for unique analysis batch.	Text(50)		

**Hudson River Baseline Monitoring Program**  
USEPA Fish Database Export Dictionary

**COMPOSITES (Data for individual fish in composite samples)**

#	Attribute Name	Description	Data Type (Size)	Units	Notes
1	FIELD_SAMPLE_ID	Sample ID for unique composite sample. Used to link individual fish in a composite with the composite information in the DESCRIPTION and Results tables.	Text(50)		
2	INDIVIDUAL_ID	ID of individual fish in composite.	Long Integer		
3	SPECIES	NYSDEC abbreviations for species of fish sample.	Text(50)		
4	SEX	Sex of individual fish sample.	Text(50)		M: male F: female U: unknown
5	AGE	Age of individual fish	Long Integer		
6	TOTAL_LENGTH_MM	Total length of fish sample for individuals (nearest mm).	Long Integer	mm	
7	WEIGHT_G	Total weight of fish sample for individuals (nearest 0.1 grams).	Single	g	
8	PREP	Indicates type of sample collected.	Text(50)		"fillet" or "whole body"
9	REMARKS	General comments or field observations at time of sample collection.	Text(255)		

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

**LOCATIONS**

#	Attribute Name	Attribute Definition	Data Type	Units	Notes
1	FIELD_SAMPLE_ID	Unique field sample ID.	Text(50)		Example: "RTN-040609-L1-C01"
2	LOCATION_NAME	Name of sampling location (e.g., Stillwater).	Text(30)		
3	PARENT_SAMPLE_ID	Blank for normal field samples. The value of "FIELD_SAMPLE_ID" that uniquely identifies the sample that was the source of this sample.	Text(50)		
4	PARTITION_PARENT_SAMPLE_ID	Field sample ID of parent sample for particulate / dissolved phase study samples.	Text(30)		
5	SAMPLE_MATRIX_CODE	Code which distinguishes between different types of sample matrix.	Text(50)		D: dissolved filtrate R: filter residue W: whole water sample
6	SAMPLE_TYPE_CODE	Code which distinguishes between different types of samples.	Text(50)		ENV: environmental sample DUP: duplicate sample FDBL: field blank
7	SAMPLE_SOURCE	This field identifies where the sample came from.	Text(10)		"Field" or "Lab"
8	SAMPLE_DATE_TIME	Date and time sample was collected.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
9	CHAIN_OF_CUSTODY	Chain of custody identifier.	Text(50)		
10	SAMPLER_INITIALS	Initials of sample collection personnel.	Text(50)		
11	SAMPLE_ARCHIVED	Indicates if a sample was archived.	Text(50)		"Yes" or "No"
12	EPA_SPLIT	Indicates if the sample was chosen as a split by the USEPA.	Text(50)		"Yes" or "No"
13	SAMPLE_TYPE	Indicates type of water sample collected.	Text(20)		"TRANSECT COMPOSITE", "CENTER CHANNEL", or "E-W COMPOSITE"
14	COMMENTS	General comments or field observations at time of sample collection.	Text(255)		

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type	Units	Notes
15	VOLUME	Indicates if sample is routine or high-volume sample for PCB analysis.	Text(15)		"ROUTINE" or "HIGH VOLUME"
16	T1	Distance from 0 (west shore) for EDI location 1.	Long Integer	ft	
17	T2	Distance from 0 (west shore) for EDI location 2.	Long Integer	ft	
18	T3	Distance from 0 (west shore) for EDI location 3.	Long Integer	ft	
19	T4	Distance from 0 (west shore) for EDI location 4.	Long Integer	ft	
20	T5	Distance from 0 (west shore) for EDI location 5.	Long Integer	ft	
21	T6	Distance from 0 (west shore) for EDI location 6.	Long Integer	ft	

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

**SWQ – Surface Water Quality Data**

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
1	TRANSECT_POINT	Transect number at which the surface water quality measurements were taken.	Long Integer		
2	FIELD_SAMPLE_ID	Field sample ID from LOCATIONS table corresponding to transect point.	Text(50)		
3	TRANSECT_SAMPLE_ID	Unique identifier for each location transect number. ID's for duplicate measurements end with "D".	Text(50)		Example: "RTN-040609-WF-T01"
4	DATE_TIME	Date and time water quality information was measured.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
5	SPCOND	Specific conductivity of water.	Single	mS/cm	
6	TEMP	Water temperature.	Single	Degrees Celsius	
7	TURB	Turbidity.	Single	NTU	
8	PH	pH of water.	Single		
9	D_O	Dissolved oxygen concentration.	Single	mg/L	
10	DEPTH	Depth from water surface that water quality information was measured.	Single	ft	
11	NOTES	General comments regarding surface water quality data.	Text(255)		

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

**RESULTS\_PCBs and RESULTS\_NONPCBs**

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
1	FIELD_SAMPLE_ID	Unique field sample ID.	Text(50)		Example: "RTN-040609-L1-C01"
2	LAB_SAMPLE_ID	Laboratory sample identifier.	Text(60)		
3	SAMPLE_TYPE_CODE	Code which distinguishes between different types of sample.	Text(25)		ENV: environmental sample DUP: duplicate sample FDBL: field blank MB: method blank LCS: laboratory control sample MS: matrix spike MSD: matrix spike duplicate
4	SAMPLE_MATRIX_CODE	Code which distinguishes between different types of sample matrix.	Text(25)		W: whole water sample D: dissolved filtrate R: filter residue
5	SAMPLE_SOURCE	This field identifies where the sample came from.	Text(10)		"Field" or "Lab"
6	SAMPLE_COMMENT	Sample comments as necessary.	Text(255)		
7	LAB_ANALYTICAL_METHOD	Laboratory analytical method name or description.	Text(50)		
8	ANALYSIS_DATE_TIME	Date of sample analysis.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
9	TOTAL_OR DISSOLVED	"T" for total (metal) concentration, "D" for dissolved or filtered (metal) concentration, or "N" for organic (or other) constituents for which neither "total" nor "dissolved" is applicable.	Text(1)		"T", "D", or "N"
10	COLUMN_NUMBER	"1C" for first column analyses, "2C" for second column analyses, or "NA" for analyses for which neither "1C" nor "2C" is applicable.	Text(5)		"1C", "2C", or "NA"
11	TEST_TYPE	Type of test.	Text(20)		"initial", "reextract", or "reanalysis".
12	CAS_RN	Chemical Abstracts Registry Number for the parameter if available.	Text(20)		
13	PARAMETER	Chemical name.	Text(60)		
14	RESULT_VALUE	Analytical result.	Double		Blank for non-detects.

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
15	RESULT_ERROR_DELTA	Error range applicable to the result value.	Double		
16	RESULT_TYPE_CODE	"TRG" for a target or regular result, and "SUR" for surrogates.	Text(10)		"TRG" or "SUR"
17	REPORTABLE_RESULT	"Yes" for results which are considered to be reportable, or "No" for non-reportable results.	Text(10)		"Yes" or "No"
18	DETECT_FLAG	"Y" for detected analytes or "N" for non-detects.	Text(3)		"Y" or "N"
19	QC_LEVEL	Status of data quality review.	Text(50)		"Verified" or "Validated"
20	RESULT_QUALIFIERS	Qualifiers assigned to samples during data verification /validation.	Text(50)		
21	ORGANIC_YN	"Y" for organic constituents or "N" for inorganic constituents.	Text(3)		"Y" or "N"
22	MDL	Method detection limit.	Double		
23	RL	Detection limit that reflects conditions such as dilution factors and moisture content.	Double		
24	QL	Concentration level above which results can be quantified with confidence.	Double		
25	RESULT_UNIT	Units of measurement for the result.	Text(15)		
26	DETECTION_LIMIT_UNIT	Units of measurement for the detection limit(s).	Text(15)		
27	RESULT_COMMENT	Result specific comments.	Text(255)		
28	QC_ORIGINAL_CONC	The concentration of the analyte in the original (unspiked) sample.	Double		
29	QC_SPIKE_ADDED	The concentration of the analyte added to the original sample.	Double		
30	QC_SPIKE_MEASURED	The measured concentration of the analyte.	Double		
31	QC_SPIKE_RECOVERY	The percent recovery calculated.	Double		
32	QC_DUP_ORIGINAL_CONC	The concentration of the analyte in the original sample.	Double		
33	QC_DUP_SPIKE_ADDED	The concentration of the analyte added to the original sample.	Double		

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
34	QC_DUP_SPIKE_MEASURED	The measured concentration of the analyte in the duplicate (for background corrected matrix spike duplicates).	Double		
35	QC_DUP_SPIKE_RECOVERY	The duplicate percent recovery calculated.	Double		
36	QC_RPD	The relative percent difference calculated.	Double		
37	QC_SPIKE_LCL	Lower control limit for spike recovery.	Double		
38	QC_SPIKE_UCL	Upper control limit for spike recovery.	Double		
39	QC_RPD_CL	Relative percent difference control limit.	Double		
40	QC_SPIKE_STATUS	Indicates whether the spike recovery was within control limits. The "*" character indicates failure; otherwise blank.	Text(20)		
41	QC_DUP_SPIKE_STATUS	Indicates whether the duplicate spike recovery was within control limits. The "*" character indicates failure; otherwise blank.	Text(20)		
42	QC_RPD_STATUS	Indicates whether the relative percent difference was within control limits. The "*" character indicates failure; otherwise blank.	Text(20)		
43	LAB_MATRIX_CODE	Code which distinguishes between different types of lab sample matrix.	Text(10)		"W"
44	ANALYSIS_LOCATION	"FI" for field instrument or probe, "FL" for mobile field laboratory analysis, or "LB" for fixed-based laboratory analysis.	Text(2)		"FI", "FL", or "LB"
45	BASIS	"Wet" for wet-weight basis reporting, "Dry" for dry-weight basis reporting, or "NA" for tests for which this distinction is not applicable.	Text(10)		"Wet", "Dry" or "NA"
46	DILUTION_FACTOR	Effective test dilution factor.	Double		
47	PREP_METHOD	Laboratory sample preparation method name or description.	Text(50)		
48	PREP_DATE_TIME	Date of sample preparation.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
49	LAB_NAME_CODE	Unique identifier of the laboratory.	Text(15)		
50	DATA_PACKAGE_LEVEL	Data package level.	Text(10)		"A", "B", or "AB"
51	PERCENT_MOISTURE	Percent moisture of the sample portion used in this test.	Double		
52	SUBSAMPLE_AMOUNT	Amount of original sample used in sample preparation.	Double		
53	SUBSAMPLE_AMOUNT_UNIT	Unit of measurement for subsample amount.	Text(15)		
54	SAMPLE_DELIVERY_GROUP	Sample delivery group.	Text(20)		
55	TEST_COMMENT	Comments about the test as necessary.	Text(255)		
56	FINAL_VOLUME	The final amount/volume of the sample, extract, or digestate after sample preparation.	Double		
57	FINAL_VOLUME_UNIT	The unit of measure that corresponds to the final volume.	Text(15)		
58	PREP_BATCH_ID	ID for unique prep batch.	Text(50)		
59	ANALYSIS_BATCH_ID	ID for unique analysis batch.	Text(50)		

**APPENDIX E**  
**USGS FLOW AND NRCC METEOROLOGICAL DATA**  
**(CD-ROM ATTACHED)**

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

**LOCATIONS**

#	Attribute Name	Attribute Definition	Data Type	Units	Notes
1	FIELD_SAMPLE_ID	Unique field sample ID.	Text(50)		Example: "RTN-040609-L1-C01"
2	LOCATION_NAME	Name of sampling location (e.g., Stillwater).	Text(30)		
3	PARENT_SAMPLE_ID	Blank for normal field samples. The value of "FIELD_SAMPLE_ID" that uniquely identifies the sample that was the source of this sample.	Text(50)		
4	PARTITION_PARENT_SAMPLE_ID	Field sample ID of parent sample for particulate / dissolved phase study samples.	Text(30)		
5	SAMPLE_MATRIX_CODE	Code which distinguishes between different types of sample matrix.	Text(50)		D: dissolved filtrate R: filter residue W: whole water sample
6	SAMPLE_TYPE_CODE	Code which distinguishes between different types of samples.	Text(50)		ENV: environmental sample DUP: duplicate sample FDBL: field blank
7	SAMPLE_SOURCE	This field identifies where the sample came from.	Text(10)		"Field" or "Lab"
8	SAMPLE_DATE_TIME	Date and time sample was collected.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
9	CHAIN_OF_CUSTODY	Chain of custody identifier.	Text(50)		
10	SAMPLER_INITIALS	Initials of sample collection personnel.	Text(50)		
11	SAMPLE_ARCHIVED	Indicates if a sample was archived.	Text(50)		"Yes" or "No"
12	EPA_SPLIT	Indicates if the sample was chosen as a split by the USEPA.	Text(50)		"Yes" or "No"
13	SAMPLE_TYPE	Indicates type of water sample collected.	Text(20)		"TRANSECT COMPOSITE", "CENTER CHANNEL", or "E-W COMPOSITE"
14	COMMENTS	General comments or field observations at time of sample collection.	Text(255)		

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type	Units	Notes
15	VOLUME	Indicates if sample is routine or high-volume sample for PCB analysis.	Text(15)		"ROUTINE" or "HIGH VOLUME"
16	T1	Distance from 0 (west shore) for EDI location 1.	Long Integer	ft	
17	T2	Distance from 0 (west shore) for EDI location 2.	Long Integer	ft	
18	T3	Distance from 0 (west shore) for EDI location 3.	Long Integer	ft	
19	T4	Distance from 0 (west shore) for EDI location 4.	Long Integer	ft	
20	T5	Distance from 0 (west shore) for EDI location 5.	Long Integer	ft	
21	T6	Distance from 0 (west shore) for EDI location 6.	Long Integer	ft	

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

**SWQ – Surface Water Quality Data**

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
1	TRANSECT_POINT	Transect number at which the surface water quality measurements were taken.	Long Integer		
2	FIELD_SAMPLE_ID	Field sample ID from LOCATIONS table corresponding to transect point.	Text(50)		
3	TRANSECT_SAMPLE_ID	Unique identifier for each location transect number. ID's for duplicate measurements end with "D".	Text(50)		Example: "RTN-040609-WF-T01"
4	DATE_TIME	Date and time water quality information was measured.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
5	SPCOND	Specific conductivity of water.	Single	mS/cm	
6	TEMP	Water temperature.	Single	Degrees Celsius	
7	TURB	Turbidity.	Single	NTU	
8	PH	pH of water.	Single		
9	D_O	Dissolved oxygen concentration.	Single	mg/L	
10	DEPTH	Depth from water surface that water quality information was measured.	Single	ft	
11	NOTES	General comments regarding surface water quality data.	Text(255)		

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

**RESULTS\_PCBs and RESULTS\_NONPCBs**

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
1	FIELD_SAMPLE_ID	Unique field sample ID.	Text(50)		Example: "RTN-040609-L1-C01"
2	LAB_SAMPLE_ID	Laboratory sample identifier.	Text(60)		
3	SAMPLE_TYPE_CODE	Code which distinguishes between different types of sample.	Text(25)		ENV: environmental sample DUP: duplicate sample FDBL: field blank MB: method blank LCS: laboratory control sample MS: matrix spike MSD: matrix spike duplicate
4	SAMPLE_MATRIX_CODE	Code which distinguishes between different types of sample matrix.	Text(25)		W: whole water sample D: dissolved filtrate R: filter residue
5	SAMPLE_SOURCE	This field identifies where the sample came from.	Text(10)		"Field" or "Lab"
6	SAMPLE_COMMENT	Sample comments as necessary.	Text(255)		
7	LAB_ANALYTICAL_METHOD	Laboratory analytical method name or description.	Text(50)		
8	ANALYSIS_DATE_TIME	Date of sample analysis.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM
9	TOTAL_OR DISSOLVED	"T" for total (metal) concentration, "D" for dissolved or filtered (metal) concentration, or "N" for organic (or other) constituents for which neither "total" nor "dissolved" is applicable.	Text(1)		"T", "D", or "N"
10	COLUMN_NUMBER	"1C" for first column analyses, "2C" for second column analyses, or "NA" for analyses for which neither "1C" nor "2C" is applicable.	Text(5)		"1C", "2C", or "NA"
11	TEST_TYPE	Type of test.	Text(20)		"initial", "reextract", or "reanalysis".
12	CAS_RN	Chemical Abstracts Registry Number for the parameter if available.	Text(20)		
13	PARAMETER	Chemical name.	Text(60)		
14	RESULT_VALUE	Analytical result.	Double		Blank for non-detects.

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
15	RESULT_ERROR_DELTA	Error range applicable to the result value.	Double		
16	RESULT_TYPE_CODE	"TRG" for a target or regular result, and "SUR" for surrogates.	Text(10)		"TRG" or "SUR"
17	REPORTABLE_RESULT	"Yes" for results which are considered to be reportable, or "No" for non-reportable results.	Text(10)		"Yes" or "No"
18	DETECT_FLAG	"Y" for detected analytes or "N" for non-detects.	Text(3)		"Y" or "N"
19	QC_LEVEL	Status of data quality review.	Text(50)		"Verified" or "Validated"
20	RESULT_QUALIFIERS	Qualifiers assigned to samples during data verification /validation.	Text(50)		
21	ORGANIC_YN	"Y" for organic constituents or "N" for inorganic constituents.	Text(3)		"Y" or "N"
22	MDL	Method detection limit.	Double		
23	RL	Detection limit that reflects conditions such as dilution factors and moisture content.	Double		
24	QL	Concentration level above which results can be quantified with confidence.	Double		
25	RESULT_UNIT	Units of measurement for the result.	Text(15)		
26	DETECTION_LIMIT_UNIT	Units of measurement for the detection limit(s).	Text(15)		
27	RESULT_COMMENT	Result specific comments.	Text(255)		
28	QC_ORIGINAL_CONC	The concentration of the analyte in the original (unspiked) sample.	Double		
29	QC_SPIKE_ADDED	The concentration of the analyte added to the original sample.	Double		
30	QC_SPIKE_MEASURED	The measured concentration of the analyte.	Double		
31	QC_SPIKE_RECOVERY	The percent recovery calculated.	Double		
32	QC_DUP_ORIGINAL_CONC	The concentration of the analyte in the original sample.	Double		
33	QC_DUP_SPIKE_ADDED	The concentration of the analyte added to the original sample.	Double		

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
34	QC_DUP_SPIKE_MEASURED	The measured concentration of the analyte in the duplicate (for background corrected matrix spike duplicates).	Double		
35	QC_DUP_SPIKE_RECOVERY	The duplicate percent recovery calculated.	Double		
36	QC_RPD	The relative percent difference calculated.	Double		
37	QC_SPIKE_LCL	Lower control limit for spike recovery.	Double		
38	QC_SPIKE_UCL	Upper control limit for spike recovery.	Double		
39	QC_RPD_CL	Relative percent difference control limit.	Double		
40	QC_SPIKE_STATUS	Indicates whether the spike recovery was within control limits. The "*" character indicates failure; otherwise blank.	Text(20)		
41	QC_DUP_SPIKE_STATUS	Indicates whether the duplicate spike recovery was within control limits. The "*" character indicates failure; otherwise blank.	Text(20)		
42	QC_RPD_STATUS	Indicates whether the relative percent difference was within control limits. The "*" character indicates failure; otherwise blank.	Text(20)		
43	LAB_MATRIX_CODE	Code which distinguishes between different types of lab sample matrix.	Text(10)		"W"
44	ANALYSIS_LOCATION	"FI" for field instrument or probe, "FL" for mobile field laboratory analysis, or "LB" for fixed-based laboratory analysis.	Text(2)		"FI", "FL", or "LB"
45	BASIS	"Wet" for wet-weight basis reporting, "Dry" for dry-weight basis reporting, or "NA" for tests for which this distinction is not applicable.	Text(10)		"Wet", "Dry" or "NA"
46	DILUTION_FACTOR	Effective test dilution factor.	Double		
47	PREP_METHOD	Laboratory sample preparation method name or description.	Text(50)		
48	PREP_DATE_TIME	Date of sample preparation.	Text(255)		MM/DD/YYYY HH:MM:SS AMPM

**Hudson River Baseline Monitoring Program**  
USEPA Water Database Export Dictionary

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
49	LAB_NAME_CODE	Unique identifier of the laboratory.	Text(15)		
50	DATA_PACKAGE_LEVEL	Data package level.	Text(10)		"A", "B", or "AB"
51	PERCENT_MOISTURE	Percent moisture of the sample portion used in this test.	Double		
52	SUBSAMPLE_AMOUNT	Amount of original sample used in sample preparation.	Double		
53	SUBSAMPLE_AMOUNT_UNIT	Unit of measurement for subsample amount.	Text(15)		
54	SAMPLE_DELIVERY_GROUP	Sample delivery group.	Text(20)		
55	TEST_COMMENT	Comments about the test as necessary.	Text(255)		
56	FINAL_VOLUME	The final amount/volume of the sample, extract, or digestate after sample preparation.	Double		
57	FINAL_VOLUME_UNIT	The unit of measure that corresponds to the final volume.	Text(15)		
58	PREP_BATCH_ID	ID for unique prep batch.	Text(50)		
59	ANALYSIS_BATCH_ID	ID for unique analysis batch.	Text(50)		

## EPA Climate/Flow Database Export Dictionary

**CLIMATE DATA**

#	Attribute Name	Attribute Definition	Data Type	Units	Notes
1	STATION_NAME	Name of climate station (e.g., GLENS FALLS MEMORIAL AP).	Text(50)		
2	STATION_ID	ID number for climate station.	Long Integer		
3	YEAR	Year of reading.	Long Integer		
4	MONTH	Month of reading.	Long Integer		
5	DAY	Day of reading.	Long Integer		
6	MAX_TEMP	Maximum daily temperature.	Text(50)	°F	*** = missing data
7	MIN_TEMP	Minimum daily temperature.	Text(50)	°F	*** = missing data
8	AVG_TEMP	Average daily temperature.	Text(50)	°F	*** = missing data
9	DEPARTURE	Departure from normal temperature.	Text(50)	°F	*** = missing data
10	HDD	Heating degree days, base 65.	Text(50)		*** = missing data
11	CDD	Cooling degree days, base 65.	Text(50)		*** = missing data
12	GDD	Growing degree days, base 50.	Text(50)		*** = missing data
13	PRECIPITATION	Daily precipitation.	Text(50)	Inches	*** = missing data; tr = trace
14	SNOW_FALL	Daily snow fall.	Text(50)	Inches	*** = missing data
15	SNOW_DEPTH	Snow depth.	Text(50)	Inches	*** = missing data

**HUDSON FLOW**

#	Attribute Name	Attribute Definition	Data Type (Size)	Units	Notes
1	DATE	Date information was collected (eastern standard time).	Date/Time		MM/DD/YYYY
2	FLOW	Daily mean flow rate.	Text(50)	Cubic feet per second (cfs).	ICE = Flow at station affected by ice.
3	QC_Code	Data-value qualification codes.	Text(5)		A - Approved for publication -- Processing and review completed. P - Provisional data subject to revision. e - Value has been estimated.

**Warning about provisional USGS flow data:**

Flow data is provisional and subject to revision.

Recent data provided by the USGS in New York -- including stream discharge, water levels, precipitation, and components from water-quality monitors--are preliminary and have not received final approval.

Most data relayed by satellite or other telemetry have received little or no review. Inaccuracies in the data may be present because of instrument malfunctions or physical changes at the measurement site. **Subsequent review may result in significant revisions to the data.**

Data users are cautioned to consider carefully the provisional nature of the information before using it for decisions that concern personal or public safety or the conduct of business that involves substantial monetary or operational consequences.

Information concerning the accuracy and appropriate uses of these data or concerning other hydrologic data may be obtained from the station manager, whose name is shown on the single station data summary pages, or from the USGS surface-water specialist in New York care of the webmaster email alias New York NWISWeb Maintainer.