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December 18, 2023

Mr. Michael Belveg, Assistant Engineer
New York State Department of Environmental Conservation
Division of Environmental Remediation, Region 7
5786 Widewaters Parkway
Syracuse, NY 13214-1867

Subject: Carrier Corporation Thompson Road Facility
Onondaga County, Syracuse, New York
Corrective Action Order – Index CO 7-20051118-4
Site Registry No. 734043

Biota Monitoring Plan – Sanders Creek Site
Response to NYSDEC Comment Letter Dated October 17, 2023

Dear Mr. Belveg:

On behalf of Carrier Corporation (Carrier), AECOM Technical Services, Inc. (AECOM), is submitting this response to the New York State Department of Environmental Conservation (NYSDEC) comment letter dated October 17, 2023, regarding the *Biota Monitoring Plan – Sanders Creek Site* (BMP; Gradient, 2023). Responses to NYSDEC comments are provided below and the revised BMP is attached.

1. NYSDEC Comment #1: *Please provide a Quality Assurance Project Plan including laboratory standard operating procedures, method quantification limits, number and type of quality control samples, performance metrics, acceptance criteria, etc. Any chosen lab will need to demonstrate the ability to analyze targets in tissue samples and include reference material in every sample delivery group. The Soxhlet extraction method is preferred over automated methodologies.*

   Response to Comment #1: As requested, a quality assurance project plan has been included as an attachment to the revised BMP.

2. NYSDEC Comment #2: *Please specify that field collection data will be provided to DEC by December 15th of the same year as collection.*

   Response to Comment #2: As requested, the date has been included under section 3.2 of the revised BMP.

3. NYSDEC Comment #3: *1.2 Site Description - Section identifies Reach 0 as an upgradient/background reach. As depicted, Reach 0 is on Carrier property and receives drainage from a previously remediated area. It is unqualified to be a background location. Relocate the background reach upstream of the Carrier property or propose an alternative background sampling location.*

   Response to Comment #3: The NYSDEC-approved Interim Corrective Measures Work Plan (ICM WP; AECOM, 2021) specifies Reach 0 as the background sampling location.

4. NYSDEC Comment #4: *Sampling Frequency - Table 2.1 Please remove the fifth row of this table as the text in this row can be misinterpreted and is not needed.*

   Response to Comment #4: As requested, the row has been removed in the revised BMP.

5. NYSDEC Comment #5: *2.2 Target Species –*

   a. *Section states biota monitoring will center on collection of Cambaridae. The typical adult Cambaridae from a stream size similar to Sanders Creek is approximately 2.5-3 inches. The report suggests needing 50-150 grams crayfish for the laboratory analysis and suggests needing a minimum of 25 adult Cambaridae sp. per sample reach for 50 grams of sample. The crayfish sampling using the parameters as stated are unlikely to be met based on comparison with other regional crayfish studies in similar streams.*

      i. *Please modify the text here and throughout the BMP to state that monitoring will include crayfish sampling but will not primarily center on crayfish sampling.*

      ii. *Please modify the report to indicate that forage fish will be the primary target of the monitoring plan.*

      iii. *Please provide how Cambaridae crayfish ID will be QA/QC'd in the field.*

      iv. *Please consider collecting all crayfish species and processing different species similar to how fish are handled.*

      v. *Please clarify how the crayfish will be processed by the laboratory.*

   b. *For the purpose of monitoring, the sampling should target a minimum of 10 samples per location and a minimum of 5 of those samples should be*

forage fish. If enough crayfish cannot be collected to make up 5 samples, forage fish should be used to make up the difference in sample number. Please emphasize in this section and throughout the report that the priority of the field effort is to meet ten samples per sampling reach.

c. *If insufficient target forage fish sp. (whatever species that is common at every sampling reach) cannot be collected to meet the sample number, then the next most common species should be utilized to fill the sample number. The priority is to collect a minimum of 5 forage fish samples within each reach. The sample number should be completed even if it is necessary to use composites of a species that is not collected at all of the other sample reaches.*

d. *All forage fish for the entire field effort should be retained until collections of all reaches are completed to be able to identify the common species among all the sample reach locations and process samples for the laboratory.*

Response to Comment #5a i and #5a ii: Per the NYSDEC-approved ICM WP, biota monitoring will include finfish, but will focus on crayfish. The content of the BMP reflects this approval and details that both crayfish and finfish will be collected, as available to be collected.

Response to Comment #5a iii: As requested, the text has been revised section 2.2.2 under bullet #6 of the revised BMP to include how the crayfish will be field identified.

Response to Comment #5a iv: As requested, the text has been revised section 2.2.2 under bullet #6 of the revised BMP.

Response to Comment #5a v: As requested, the text has been revised section 2.2.2 under bullet #6 of the revised BMP to include how the crayfish will be processed by the laboratory.

Response to Comment #5b and #5c: As requested, the text and Table 2.2 has been revised in section 2.2.1 of the revised BMP.

Response to Comment #5d: As requested, the text has been revised section 2.2.2 under bullet #6 of the revised BMP.

6. NYSDEC Comment #6: *Section 2.2 Sampling Time, Location, and Method - Please list and specify that DEC guidance "Procedures of Collection and Preparation of Aquatic Biota for Contaminant Analysis will be adhered to.*

Response to Comment #6: As requested, the reference to the NYSDEC guidance has been included in sections 1 and 2 of the revised BMP.

7. NYSDEC Comment #7: *Section 2.2.2 Sampling Method - Please consider hand collection and baited traps for the collection of crayfish as the increased variety of collection methods are likely to increase the chances of successfully meeting the required sample numbers. In the years that follow this initial sampling event, the field effort can focus on the most successful collection method(s).*

   Response to Comment #7: As requested, the text has been revised in section 2.2.2 under bullet #1 of the revised BMP.

Should you have any questions, please contact me at 919 461-1194.

Yours sincerely,

Peter Hollatz
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References

AECOM 2021. *Interim Corrective Measure Work Plan*. AECOM, April 2021.

Gradient 2023. *Biota Monitoring Plan – Sanders Creek Site.* Gradient, July 2023.

Attachments

Biota Monitoring Plan (Revision 01)

cc: Gary Priscott, NYSDEC
Don Sorbello, Carrier Corporation

Biota Monitoring Plan (Revision 01)

Sanders Creek Site
Thompson Road, Syracuse, NY

Prepared for
Carrier Corporation
Thompson Road
Syracuse, NY 13206

December 18, 2023

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Abbreviations

BMP	Biota Monitoring Plan
$C_{lipid\text{-}normalized}$	Lipid-normalized Total PCB Concentration (µg/g-lipid)
C_{tissue}	Total Body PCB Concentration for a Given Sample (µg/g)
DER	Division of Environmental Remediation
FSAP	Field Sampling and Analysis Plan
HASP	Health and Safety Plan
ICM	Interim Corrective Measures
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NYSDEC	New York State Department of Environmental Conservation
ORP	Oxidation and Reduction Potential
PCB	Polychlorinated Biphenyl
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RG	Remedial Goal
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
US EPA	United States Environmental Protection Agency
WP	Work Plan
%lipid	Percentage Lipid Concentration for a Given Composite Tissue Sample

1 Introduction

Gradient has prepared this Biota Monitoring Plan (BMP) to monitor the performance and effectiveness of a planned Remedial Action in a section of Sanders Creek. Sanders Creek is located in the town of DeWitt, Onondaga County, New York (Figure 1.1). The portion of Sanders Creek beginning north of Carrier's Thompson Road facility (Carrier facility) and continuing downstream to the confluence with the South Branch of Ley Creek is considered the Site.

An Interim Corrective Measures (ICM) Work Plan (WP) was prepared by Carrier Corporation (Carrier) in response to the New York State Department of Environmental Conservation (NYSDEC) Corrective Action Order – Index Number CO 7-20051118-4 dated January 4, 2006 (AECOM, 2021). The ICM WP outlines the Remedial Action that will be implemented to achieve the remedial criteria agreed to by NYSDEC for polychlorinated biphenyls (PCBs) in the Sanders Creek sediments and immediate floodplains which support achieving the Remedial Goal (RG) as specified by the Corrective Action Order:

> The remedial goal/criteria for Sanders Creek is that monitoring of resident aquatic biota assures that PCB concentrations are 0.1 parts per million (ppm) or less in the relevant portions of the creek. (AECOM, 2021).

This BMP was prepared considering the following state and federal technical guidance documents:

- NYSDEC, 2002. "Procedures for Collection and Preparation of Aquatic Biota for Contaminant Analysis (Draft)."
- NYSDEC, 2010. "Division of Environmental Remediation (DER)-10: Technical Guidance for Site Investigation and Remediation."
- NYSDEC, 2013. "Freshwater Fisheries Management."
- NYSDEC, 2020. "Quality Assurance Project Plan: Rapid Assessment Surveys."
- NYSDEC, 2021. "Standard Operating Procedures: Biological Monitoring of Surface Waters in New York State."
- US EPA, 1999. "Rapid Bioassessment Protocols for Use in Wadable Streams and Rivers – Periphyton, Benthic Macroinvertebrates, and Fish."
- US EPA, 2000a. "Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Volume 1 Fish Sampling and Analysis." Third Edition.
- US EPA, 2008. "Using Fish Tissue Data to Monitor Remedy Effectiveness."

1.1 Purpose, Objective, and Scope

The purpose of a monitoring plan is to "define the measures for monitoring the performance and effectiveness of the remedy at the Site" (AECOM, 2021). The objective of this BMP is to monitor the performance and effectiveness of a planned Remedial Action in regards to the RG of achieving 0.1 mg/kg PCB concentrations or less in biota tissues at the Site. Specifically, the BMP will be conducted to document

PCB concentrations in crayfish and finfish tissues, to allow evaluation of compliance with the RG, and to track changes in tissue concentrations following remedy implementation.

The performance and effectiveness of the Remedial Action will be evaluated by establishing a new baseline biota PCB tissue dataset prior to remediation, collecting post-remedy tissue samples, and analyzing temporal and spatial trends in PCB tissue concentrations.

As detailed in the NYSDEC-approved ICM WP (AECOM, 2021), crayfish and finfish will both be targeted for tissue monitoring since they are expected to be present at the Site in sufficient and reliable quantities to allow for long-term biomonitoring. In addition, crayfish have limited home ranges, which is useful when monitoring tissue concentrations most likely associated with localized exposures (AECOM, 2021).

1.2 Site Description

Sanders Creek is classified as a Class C, Standard C water under the NYSDEC Protection of Waters Regulatory Program (AECOM, 2021). Class C is defined as: "The best usage of Class C waters is fishing. These waters will be suitable for fish, shellfish, and wildlife propagation and survival. The water quality will be suitable for primary and secondary contact recreation, although other factors may limit the use for these purposes." The Site consists of the portion of Sanders Creek beginning north of the Carrier facility and continuing downstream to the confluence with the South Branch of Ley Creek. Sanders Creek flows toward the west through wooded and developed areas and is connected through a series of culverts. The Site has been divided into the following seven reaches based on the presence of culverts and property boundaries (Figure 1.2). In addition, an upgradient reach of Sanders Creek will be used to define background conditions.

- Reach 0 (background/upgradient reach): From Kinne Street to the culvert under Telergy Parkway.
- Reach 1: Between culvert under Telergy Parkway to the next downstream culvert (Culvert 1).
- Reach 2: Between Culvert 1 to the culvert under Thompson Road.
- Reach 3: Between culvert under Thompson Road to the next downstream culvert (Culvert 2).
- Reach 4: Between Culvert 2 to the Carrier property boundary.
- Reach 5: Between the Carrier property boundary to the culvert under Old Court Street Road.
- Reach 6: Between culvert under Old Court Street Road to the next downstream culvert at a private road (Culvert 3).
- Reach 7: Between the culvert at Deere Road to the confluence of South Branch of Ley Creek.

1.3 Report Organization

The remainder of this BMP is organized into three sections based on the elements outlined in Section 6.2 of the ICM WP (AECOM, 2021):

- Section 2 (Sampling Approach and Methods) describes the target species, sampling methods, and analytical methods.
- Section 3 (Data Evaluation and Reporting) describes the data analyses and reporting methods. This section also discusses the approach that may be used to adjust the BMP methods for future sampling events based on past data and analyses.

- References provides a list of references used cited in the BMP.

2 Sampling Approach and Methods

This section describes the sampling approach and methods that will be used, following the information provided in Sections 6.2.2 and 6.2.3 of the ICM WP. The objective of the proposed sampling is to collect biota tissue samples, ideally during the same time of year and under similar stream flow conditions for each sampling event (US EPA, 2008). Tissue samples should have enough mass to reliably assess PCB concentration trends. The following sections outline the sampling frequency, target species, recommended sampling times and locations, sampling methods, and analytical methods, following applicable United States Environmental Protection Agency (US EPA) guidance for biota sampling (US EPA 2000a, 2008) and NYSDEC's "Procedures for Collection and Preparation of Aquatic Biota for Contaminant Analysis (Draft) (2002).

2.1 Sampling Frequency

As per the ICM WP (AECOM, 2021), a baseline sampling event will be conducted to document PCB concentration in biota tissue prior to the Remedial Action. While prior biota samples have been collected from the Site, these data were collected more than 10 years ago.

Once the Remedial Action is implemented, additional sampling will take place 1, 3, and 5 years post remedy (Table 2.1). Results of the different sampling events will be used to evaluate temporal and spatial trends of PCB tissue residue concentrations and potentially recommend changes to the sampling approach for future planned sampling events. Data collected during the 4th sampling event (5 years post remedy) will be used to evaluate whether further monitoring is needed.

Table 2.1. Sampling Events and Objectives

Sampling Event	Schedule	Objective
Event 1	Prior to remediation[a]	Establish baseline conditions
Event 2	1 year post remedy	Interim samping
Event 3	3 years post remedy	Interim sampling
Event 4	5 years post remedy	Interim/final sampling

Note:
(a) Schedule subject to securing access agreeements with non-Carrier owned properties.

2.2 Target Species

As detailed in the NYSDEC-approved ICM WP (AECOM, 2021), crayfish and finfish will be included in the biota monitoring.

Crayfish biota monitoring will center on the collection of *Cambaridae* crayfish because they are a key indicator species and have been routinely used for biomonitoring (NYSDEC, 2021). *Cambaridae* crayfish were abundant in Sanders Creek based on a 2006 biota sampling event (Ensafe, 2007; AECOM, 2021). Crayfish have the ability to repopulate areas relatively quickly, and due to their limited foraging range provide information on location-specific exposures. Crayfish primarily feed on aquatic plants, macroinvertebrates, and small fish (Lui, 2013; Pappas, 2002; AECOM, 2021). Crayfish are also important prey for higher trophic-level wildlife and allow for the evaluation of risks through food chain exposures.

Crayfish from the *Cambaridae* family include multiple species that are present at the Site and surrounding area. Crayfish begin their life as fertilized eggs attached to the underside of the female's abdomen from early March to late May. After hatching, juvenile crayfish will undergo several molts before detaching from the female (Dunoyer, 2016; Lui, 2013; Pappas, 2002). Crayfish continue their growth through periodic molting, shedding their old exoskeleton and growing a new exoskeleton that hardens. Crayfish create burrows in the sediment for shelter. During molting, they will seek refuge under rocks, in burrows, or within wood debris (Dunoyer, 2016; Lui, 2013; Pappas, 2002). Medium-to-large adult crayfish are typically between 10 and 150 mm in length[1] (Bouchard, 2004; Lui, 2013; Pappas, 2002) and can live up to three to six years.[2]

Finfish will also be sampled for tissue analysis. A total of 64 species of fish are known to be present in streams throughout the Syracuse, NY region and have the potential of occurring at the Site (Ensafe, 2007). As part of a sampling event in November 2006, finfish samples were collected from three sampling locations at the Site[3] to determine PCB concentrations in fish (Figure 4 in Ensafe, 2007). During this sampling event, a total of 308 individual fish were captured using a backpack electro shocker and dip net. The collected fish belonged to the following seven species (abundance included as a percentage): creek chub (53.5%), longnose dace (28.9%), white sucker (8.7%), pumpkinseed sunfish (6.8%), fathead minnow (1.3%), largemouth bass (0.4%), and bullhead catfish (0.4%). Creek chub was the most common species collected during this sampling event, followed by longnose dace. Both species, along with white sucker and pumpkinseed sunfish were collected at all three sampling stations. The most commonly collected species, creek chub, was selected as the target species for residue analyses in the 2006 sampling event (Ensafe, 2007).

Given the abundance of creek chub collected from all sampling locations during the 2006 sampling event, creek chub is the proposed target finfish species in this BMP. However, at the discretion of the field sampler and based on the species composition at the time of sampling, different and/or additional fish species may be targeted. For example, longnose dace and creek chub are both freshwater minnow species of similar size known to be present at the Site. As result, longnose dace could be selected as an alternate to creek chub based on the observed relative abundance between both species at the time of sampling. Creek chub are ubiquitous minnows that inhabit streams and lakes, including headwaters and upland lakes, and are tolerant of degraded sites. The native range of this species extends through all of New York's mainland watersheds, but it is not present on Long Island (Carlson *et al*., 2016). Typical adult creek chubs have total body lengths ranging from 12-18 cm and can live up to 3-8 years in the wild. These carnivorous fish consume plant matter, aquatic insects, small fish, amphibians, crayfish, and mollusks (Anderson, 2014). They are also prey for larger predatory fish, such as smallmouth bass and larger creek chubs. Creek chubs reach sexual maturity at the age of 3 years and mate from mid-spring to early summer. They are generally very mobile with an average maximum range of 130-195 m (Anderson, 2014).

2.2 Sampling Time, Location, and Method

NYSDEC (2021) recommends collecting macroinvertebrates between July and September using kick nets. Sampling during the spring is not recommended due to the high number of naidid worms present. A US EPA-recommended sampling time frame for crayfish was not identified. US EPA (2008) recommends fish

[1] Body length is the distance from the tip of the rostrum to the tip of the telson (US EPA, 2000a).
[2] Rusty crayfish (*Orconectes rusticus*) are found in New York and can live to be 3- 4 years old in the wildlife (Pappas, 2002). Devil crawfish (*Cambarus diogenes*) are also found in the area and can live up to 3 years in the wild (Lui, 2013).
[3] Station 1 of the 2006 sampling was the area between Court Street and 300 feet downstream west of Court Street, which corresponds to a portion of Reach 6. Station 2 was the area between Thompson Road to Court Street, which corresponds to Reaches 3, 4, and 5. Station 3 is the area between Thompson Road and 800 feet upstream of the road, which corresponds to Reach 2.

tissue sampling during late summer to early fall when lipid content is typically the highest and lipid-soluble contaminants, such as PCBs, are most likely to accumulate in lipid-rich tissues. US EPA does not recommend fish sampling during the spring when lipid content is low, as well as around spawning season (2-4 weeks before and after) to reduce variability. Given that crayfish seek shelter in their burrows when the water temperature is cold, sampling success is expected to be greater at times of the year when the water temperature is higher (Dunoyer, 2016).

Based on the above, sampling will occur in late summer to early fall. Care will be taken to ensure that tissue data is collected under similar conditions between the different sampling events. All field activities will follow the Quality Assurance Project Plan (QAPP – see Attachment A), Field Sampling and Analysis Plan (FSAP), and Site-specific Health and Safety Plan (HASP) to be prepared prior to the field activities. Work on Carrier-owned and off-Site properties will be conducted in coordination with Carrier and property owners. Field activities will include Site meetings, mobilization, health and safety monitoring, and decontamination.

2.2.1 Number of Samples and Sample Nomenclature

As per the ICM WP (AECOM, 2021), sample locations will correspond to each of the eight reaches described in Section 1.2 (Figure 1.2). Samples from Reach 0 will represent background conditions, while samples from Reaches 1-7 will represent Site conditions. The goal is to collect a minimum of 10 samples per location and a minimum of five of those samples will be finfish samples and up to five crayfish samples. If enough crayfish cannot be collected to make up five samples at a location, then forage fish will be used to make up the difference in sample number. If sufficient samples of the target finfish species (whatever finfish species is the most common at every sampling reach) cannot be collected to meet the sample number, then the next most common finfish species will be utilized to fill the sample number. Since the priority is to collect a minimum of five finfish samples within each reach, the sample number will be completed even if it is necessary to use composites of a species that is not collected at all of the other sample reaches. The sampling objective for each sampling event is to collect enough tissue biomass in each reach to support the analytical requirements (Belveg, 2019). In addition, quality assurance/quality control (QA/QC) samples will be collected. Specifically, every 20 samples collected in the field will require additional volume for a matrix spike (MS)/matrix spike duplicate (MSD) sample. Table 2.2 presents the number of samples to be collected from each reach for each sampling event.

Table 2.2 Sample Objectives for Each Sampling Event

Location	Number of Composite Samples[a]		Total
	Finfish	Crayfish	
Reach 0	≥5	≤5	≥10
Reach 1	≥5	≤5	≥10
Reach 2	≥5	≤5	≥10
Reach 3	≥5	≤5	≥10
Reach 4	≥5	≤5	≥10
Reach 5	≥5	≤5	≥10
Reach 6	≥5	≤5	≥10
Reach 7	≥5	≤5	≥10
QA/QC Samples[b]	≥2	≤ 2	≥4
Total	**≥42**	**≤ 42**	≥84

Notes:
MS/MSD = Matrix Spike/Matrix Spike Duplicate; QA/QC = Quality Assurance/Quality Control.
(a) Finfish - a minimum of five composite samples will be collected from each reach. Crayfish - up to five composite samples will be collected from each reach.
(b) 1 MS/MSD for every 20 samples.

Composite tissue samples should be labeled to include the sampling year, species (two letter code for the various species: CR for crayfish, CC for creek chub), reach number, and composite letter. For example, the first composite of crayfish collected in 2024 from Reach 4 would be labeled as 24-CR-R4-CompA (Sample year-species-Reach#-composite letter). QA samples will be labeled as a separate sample number and will be collected in reaches with abundant captured individuals. Table 2.3 presents an example table of Sample IDs for composite samples to be collected during the 2024 sampling event.

Table 2.3 Proposed Composite Sample ID Labels for the 2024 Baseline Biota Sampling Event

Sample Year	Reach #	Composite Sample #	Crayfish[a] Composite Sample ID	Finfish[b] Composite Sample ID
2024	0	A	24-CR-R0-CompA	24-CC-R0-CompA
2024	0	B	24-CR-R0-CompB	24-CC-R0-CompB
2024	0	C	24-CR-R0-CompC	24-CC-R0-CompC
2024	0	D	24-CR-R0-CompD	24-CC-R0-CompD
2024	0	E	24-CR-R0-CompE	24-CC-R0-CompE
2024	1	A	24-CR-R1-CompA	24-CC-R1-CompA
2024	1	B	24-CR-R1-CompB	24-CC-R1-CompB
2024	1	C	24-CR-R1-CompC	24-CC-R1-CompC
2024	1	D	24-CR-R1-CompD	24-CC-R1-CompD
2024	1	E	24-CR-R1-CompE	24-CC-R1-CompE
2024	2	A	24-CR-R2-CompA	24-CC-R2-CompA
2024	2	B	24-CR-R2-CompB	24-CC-R2-CompB
2024	2	C	24-CR-R2-CompC	24-CC-R2-CompC
2024	2	D	24-CR-R2-CompD	24-CC-R2-CompD
2024	2	E	24-CR-R2-CompE	24-CC-R2-CompE
2024	3	A	24-CR-R3-CompA	24-CC-R3-CompA
2024	3	B	24-CR-R3-CompB	24-CC-R3-CompB
2024	3	C	24-CR-R3-CompC	24-CC-R3-CompC
2024	3	D	24-CR-R3-CompD	24-CC-R3-CompD
2024	3	E	24-CR-R3-CompE	24-CC-R3-CompE
2024	4	A	24-CR-R4-CompA	24-CC-R4-CompA
2024	4	B	24-CR-R4-CompB	24-CC-R4-CompB
2024	4	C	24-CR-R4-CompC	24-CC-R4-CompC
2024	4	D	24-CR-R4-CompD	24-CC-R4-CompD
2024	4	E	24-CR-R4-CompE	24-CC-R4-CompE
2024	5	A	24-CR-R5-CompA	24-CC-R5-CompA
2024	5	B	24-CR-R5-CompB	24-CC-R5-CompB
2024	5	C	24-CR-R5-CompC	24-CC-R5-CompC
2024	5	D	24-CR-R5-CompD	24-CC-R5-CompD
2024	5	E	24-CR-R5-CompE	24-CC-R5-CompE
2024	6	A	24-CR-R6-CompA	24-CC-R6-CompA
2024	6	B	24-CR-R6-CompB	24-CC-R6-CompB
2024	6	C	24-CR-R6-CompC	24-CC-R6-CompC
2024	6	D	24-CR-R6-CompD	24-CC-R6-CompD
2024	6	E	24-CR-R6-CompE	24-CC-R6-CompE
2024	7	A	24-CR-R7-CompA	24-CC-R7-CompA
2024	7	B	24-CR-R7-CompB	24-CC-R7-CompB
2024	7	C	24-CR-R7-CompC	24-CC-R7-CompC
2024	7	D	24-CR-R7-CompD	24-CC-R7-CompD
2024	7	E	24-CR-R7-CompE	24-CC-R7-CompE

Notes:

(a) For crayfish, all composite sample IDs will have the same two letter species code of CR for crayfish. Each individual crayfish within a composite sample will be identified as shown in Table 2.4.

(b) For finfish, the composite sample IDs will have the same two letter species code of CC for creek chub. Each individual finfish within a composite sample will be identified as shown in Table 2.4. At the discretion of the field sampler and based on the species composition during sampling, different and/or additional fish species may be targeted. Any fish composite samples that are collected for different finfish species will follow the same sample ID convention (*i.e.*, WS = White Sucker; LD = Longnose Dace).

2.2.2 Sampling Method

Sampling for crayfish and finfish will be conducted using a similar method for the background and Site. Within each reach, the following procedures will be followed:

1. Within each reach, a location and sampling method (*i.e.*, electro fishing, kick net, seine net, hand collection, and baited traps)[4] will be selected based on field conditions. Locations and sampling methods may be determined during a separate field event prior to sampling.
2. Within each reach, the sampler will record field notes regarding the sampling date/time, weather conditions, description of the sampling area/locations including substrate, the presence/absence of wildlife. A picture of the sampling location and surrounding environment will be collected. An example field data sheet that NYSDEC (2021) uses for biomonitoring is provided in Attachment B.
3. Prior to sampling within a reach, a surface water measurement will be collected using a water quality meter, while avoiding disturbing the sediment, and recorded onto the field data sheet (Attachment B) or electronically logged. Water temperature, specific conductivity, turbidity, dissolved oxygen, oxidation and reduction potential (ORP), and pH will be measured at each sample station where samples will be collected. Water depth, presence of vegetation, and rate of stream flow at the sampling location will also be documented.
4. Once field measurements and visual inspections are recorded, samples will be collected using the biota sampling method identified under step 1. Kick net sampling is expected to be the default method for crayfish sampling, whereas electro fishing is expected to be the default method for the collection of finfish. As per NYSDEC (2021) guidance, the ideal sampling location for kick net sampling has a water depth of at least 1 meter and a current speed of ≥ 40 cm/sec. Sampling using kick net requires laying a net downstream of the sampling area and then disturbing the substrate to dislodge macroinvertebrates from their habitat to be swept into the net by the current. Electro fishing will be conducted using a backpack electro shocker or equivalent that is operated in an upstream direction with a second person collecting shocked fish by using dip nets. A combination of sampling methods may be used to ensure adequate sample volume. Similarly, multiple locations within a reach may be sampled to ensure adequate sample volume. If multiple locations are sampled and those locations are not proximate to one another, then additional data will be collected at each locations consistent with steps 2 and 3.
5. Captured animals from each sampling attempt will be combined into a bucket or deep walled pail. The sampler will note the number of individuals caught.
6. To the extent logistically possible, captured animals will be retained until collections of all reaches are completed to be able to identify common species among all the sample reach locations and inform compositing and processing samples for laboratory submission. Captured animals will be rinsed with creek water prior to collecting species, size, and observational data. A field identification key for expected crayfish in New York will be used by a freshwater macroinvertebrate taxonomist to identify the crayfish specimens. All captured animals will be identified at the species level, and recorded. Crayfish and finfish will be weighed using a digital scale with 0.1 g accuracy, sexed if possible, and measured using a caliper or a fish measuring board for larger fish with 1 mm accuracy. The maximum body length for a crayfish is the length between the rostrum and telson (US EPA, 2000a). For finfish, the maximum body length is the distance between the anterior-most part of the fish to the tip of the longest caudal fin ray (US EPA, 2000a). Information regarding the activity level and appearance will be noted on a field sheet similar to the

[4] In the years that follow the initial sampling event, the field effort will focus on the most successful collection method(s).

example presented in Table 2.4. Any abnormalities or discolorations will be recorded and photographed. For finfish, any abnormalities, such as fin erosions, skin ulcers, skeletal anomalies, tumors, or the presence of fish parasites, will be noted and photographed (US EPA, 2000a). Crayfish with cracked or damaged exoskeletons during the sampling process or animals that are not fully intact will be noted and discarded (US EPA, 2000a).

Refer to the laboratory preparation of fish tissue standard operating procedure (SOP) in the QAPP (Attachment A) for how the samples will be processed. The process will be the same for crayfish.

7. Captured individuals will be sorted by species and size into small, medium, and large individuals to ensure composite samples are composed of similarly sized individuals to minimize variability. When selecting samples for lab analysis, preference will be given to composites made up of medium or larger individuals of similar size as PCBs bioaccumulate over time. Within each reach, a minimum of 10 composite samples (minimum of 50 g composite samples[5]) will be prepared with at least five individuals per composite sample, as described in Section 2.2.1. US EPA (2008) recommends a minimum of five individuals per composite to determine that post remedy concentrations have decreased by at least 50% with a confidence level of 90-95%. Reaches that have abundant individuals captured will be used for QC samples. Samples from which MS/MSD samples will be analyzed should include three times the minimum weight requirement (minimum of 150 g composite sample). Once the tissue weight requirement is reached, individuals selected for composite analysis will be photographed together, wrapped individually in tin foil and labeled (see Table 2.3 for recommended sample nomenclature), bagged together, and placed on ice to maintain a 4 °C temperature (US EPA 2000a). Field notes will indicate which individuals were composited together for analysis (See Table 2.4). All remaining individuals that are not selected will be noted in the field notes and released back into the water.

[5] Larger individuals that meet the minimum 50 g sample weight requirement will still be composited with similar size species for analysis in order for these samples to be compared to other composite samples.

Table 2.4 Proposed Individual Sample ID Labels and Field Notes for the 2024 Baseline Biota Sampling Event (example)

Reach	Sampling Method[a]	Date	Species	Individual Sample ID[b]	Weight (g)	Length (mm)	Relative Size	Abnormalities	Activity	Composite Sample ID
R4	KN	7/8/24	Crayfish	24-CR-R4-1	13.0	130	Large	None	Active	24-CR-R4-Comp A
R4	KN	7/8/24	Crayfish	24-CR-R4-2	11.9	120	Large	None	Active	24-CR-R4-Comp A
R4	KN	7/8/24	Crayfish	24-CR-R4-3	12.6	128	Large	Slight discoloration on left claw	Active	24-CR-R4-Comp A
R4	KN	7/8/24	Crayfish	24-CR-R4-4	14.2	140	Large	None	Slow	24-CR-R4-Comp A
R4	KN	7/8/24	Crayfish	24-CR-R4-5	12.2	126	Large	None	Active	24-CR-R4-Comp A
R4	KN	7/8/24	Crayfish	24-CR-R4-6	9.6	110	Medium	None	Active	24-CR-R4-Comp B
R4	KN	7/8/24	Crayfish	24-CR-R4-7	9.5	112	Medium	None	Active	24-CR-R4-Comp B
R4	KN	7/8/24	Crayfish	24-CR-R4-8	13.7	133	Large	Cracked	Active	Discarded
R4	KN	7/8/24	Bullfrog	N/A	N/A	N/A	Large	None	Active	Released
R4	KN	7/8/24	Crayfish	24-CR-R4-10	10.3	114	Medium	None	Active	24-CR-R4-Comp B
R4	KN	7/8/24	Crayfish	24-CR-R4-11	8.2	107	Medium	None	Active	24-CR-R4-Comp B
R4	KN	7/8/24	Crayfish	24-CR-R4-12	8.7	110	Medium	None	Active	24-CR-R4-Comp B
R4	EF	7/8/24	Creek chub	24-CC-R4-1	1.9	5	Small	Torn dorsal fin, parasites observed	Slow	24-CC-R4-Comp A
R4	EF	7/8/24	Creek chub	24-CC-R4-2	2.0	5	Small	None	Active	24-CC-R4-Comp A
R4	EF	7/8/24	Creek chub	24-CC-R4-3	2.2	5.2	Small	None	Active	24-CC-R4-Comp A
R4	EF	7/8/24	Creek chub	24-CC-R4-4	2.4	5.5	Small	None	Active	24-CC-R4-Comp A
R4	EF	7/8/24	Creek chub	24-CC-R4-5	1.6	4.8	Small	None	Active	24-CC-R4-Comp A
R4	EF	7/8/24	Catfish	24-CF-R4-1	N/A	N/A	Medium	Pea size tumor near mouth	Active	Released
R4	EF	7/8/24	White sucker	24-WS-R4-1	N/A	N/A	Medium	None	Active	Released

Notes:

N/A = Not Applicable.

(a) Sampling methods will be identified using a two-letter acronym, *e.g.*, KN = Kick Net; EF = Electro Fishing.

(b) Individual IDs will consist of: sampling year, a two-letter code based on species (CR = Crayfish; CC = Creek Chub; WS = White Sucker; LD = Longnose Dace; CF = Catfish), the reach number, and the individual sample number. Five individual IDs will be assigned to create a composite sample as shown in the last column (*e.g.*, 24-CR-R4-Comp A is a crayfish composite collected in Reach 4 during 2024 and consists of individual samples 24-CR-R4-1 through 24-CR-R4-5).

2.2.3 Decontamination Procedures

Prior to moving to a new reach for sampling, all shared materials (*e.g.*, nets, traps, calipers, fish measuring boards, scales) will be rinsed with creek water to minimize cross contamination between reaches. All used disposable materials (*i.e.*, safety gloves, foil) will be discarded properly.

2.4 Laboratory Analysis, Data Deliverables, and Data Validation

The field samples along with QC samples (MS/MSD) and temperature blanks will be placed in coolers with ample space to avoid crushing crayfish. Samples will need to be shipped within 24 hours of sampling if preserved with wet ice or within 48 hours if preserved with dry ice (US EPA, 2000a). All samples will be sent to a qualified laboratory/ies based upon credentials for the required analyses.

Whole body tissue samples will be analyzed for individual PCB aroclors (Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260) using US EPA Method 8082 and a minimum reporting limit of 0.09 microgram per gram (µg/g) (NYSDEC, 2021). In addition, samples will be analyzed for lipids using Method 3540C or 3545C, and percent moisture (AECOM, 2021). Since PCBs bioaccumulate in the lipid, the amount of lipid in the samples will aid in calculating the lipid-normalized concentration to be used in the data analysis, further discussed below. The laboratory will provide tabulated results of all samples in electronic data deliverables. The laboratory will also provide additional QC information such as the laboratory volume for analysis for method blanks, instrument blanks, laboratory duplicates, and laboratory control samples. Summaries for QC data and associated raw data generated in support of the reported results (including instrument calibration) will be included in the laboratory reports and reviewed during data validation.

All analyzed data will undergo data validation. US EPA's "Guidance for Data Quality Assessment: Practical Methods for Data Analysis" (US EPA, 2000b) addresses data quality criteria and performance specifications for decision making. All data received from the analytical laboratories will be reviewed and validated according to the most current versions of the US EPA Region II data validation SOPs, US EPA Contract Laboratory Program National Functional Guidelines, and/or QC limits established within the QAPP or laboratory-derived acceptance limits. The following information will be reviewed, as applicable:

- Data package completeness
- Case narrative
- Samples receipt, condition, and holding time
- Mass spectrometer tuning
- Instrument calibrations (initial calibrations and continuing calibration verification)
- Internal standard response (for analysis by mass spectrometer only)
- Blank contamination (Laboratory) and Field Blanks (if applicable)
- Laboratory control samples and/or matrix spike blanks
- Surrogate spike or deuterated monitoring compound recoveries
- Matrix spike and matrix spike duplicate recoveries
- Analyte identification and quantitation

As part of the data validation, data qualification flags may be applied to some sample results when considering the data usability (data quality, data qualification, data preparation, reporting limits, and data adequacy):

U	The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration
UJ	The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.
C	This qualifier applies to results when the identification has been confirmed by Gas Chromatography/Mass Spectrometer (GC/MS).
X	This qualifier applies to results when GC/MS analysis was attempted but unsuccessful.

Data qualifiers applied during the data validation or reported by the laboratory will be included in the final dataset. Data quality interpretations will be summarized in data validation reports and reviewed before data are finalized.

Following data validation, all data will undergo a data usability assessment. The following issues will be considered in the data usability assessment: data quality, data qualifications, data preparation, reporting limits, and data adequacy. All rejected data ("R" qualifier), as assigned by the laboratory or data validator, are anticipated to be excluded.

3 Data Analysis and Reporting

All field and validated analytical data will be compiled into a database. This section discusses the type of data analyses that will be conducted, describes the required content of the biomonitoring reports, and describes approaches that may be used to adjust BMP for future sampling events.

3.1 Data Evaluation Methods

Two different types of data will be collected during implementation of the BMP: field data and analytical data. Field data consists of data collected at the time of sampling to help understand field conditions and assist with the interpretation of the analytical results. Analytical data provide information on the PCB tissue residue concentrations and lipid content of the biota tissue samples collected. The evaluations for each type of data are presented below.

3.1.1 Field Data

During field sampling, the following information will be recorded, as discussed in Section 2.2, and used to supplement the finding from the analytical results.

- Sampling day and time, weather
- Sampling location information
- Water quality parameters
- Number of sampling attempts by sampling method
- Number of individuals captured for each sampling attempt
- Length and weight of individuals (including those not shipped for analysis)
- Observations of activity level and abnormalities

3.1.2 Analytical Data

Laboratory PCB data will be presented as individual aroclors. Therefore, the aroclors will be summed to calculate total PCB concentrations. In these calculations, detected concentrations of each aroclor will be summed. If an aroclor is less than the reporting limit, then a concentration of 0 will be used for that aroclor in the summation. In cases where all the aroclors are not detected, the maximum analytical reporting limit among the different aroclors will be used to represent the sample. This approach is common practice and avoids inflating the total PCB concentrations by summing individual aroclor reporting limits.

PCBs bioaccumulate in the lipids of animal tissue and consequently, samples with high lipid content may have higher PCB concentrations. Therefore, sample results will also be adjusted to calculate lipid normalized total PCB tissue concentrations using the following equation:

$$C_{lipid-normalized} = \frac{C_{tissue}}{\%lipid}$$

where:

$C_{lipid\text{-}normalized}$ = Lipid-normalized total PCB concentration (µg/g-lipid);
C_{tissue} = Total body PCB concentration for a given sample (µg/g); and
%lipid = Percentage lipid concentration for a given composite tissue sample.

The individual aroclor, calculated total PCB, and lipid-normalized total PCB tissue concentrations will be used in the data analyses.

3.1.3 Data Analyses

For each sampling event, the following metrics will be calculated and tabulated separately for crayfish and finfish:

- The length and frequency distribution among individuals of each target species captured from each reach (NYSDEC, 2013).
- Summary statistics of total PCB concentrations, %lipid, and lipid-normalized total PCB concentrations by reach (*e.g.*, number of samples, number detected, minimum detected, average,[6] maximum detected, number of samples exceeding RG).
- Aroclor contributions by reach (% contribution of different aroclors to the overall tissue concentrations).

These data will be used to identify temporal (*e.g.*, using graphs) and spatial (*e.g.*, using maps) trends and to evaluate potential differences between crayfish and finfish. The data collected from individual reaches will be compared to samples collected from the background reach by species to identify statistically significant differences using appropriate quantitative statistical methods informed by the data distribution (*e.g.*, parametric, non-parametric). In addition, tissue concentration data collected from all reaches, including from the background reach, will be compared to the RG. Starting from the 2nd sampling event, statistical analyses will also be conducted to evaluate differences between sampling events and identify potential temporal trends in the data. Finally, the data will be analyzed to identify any potential correlations between PCB tissue concentration and various other parameters such as physical characteristics of the individuals (*i.e.*, length or weight) and species.

3.2 Reporting

Biomonitoring reports will be submitted to NYSDEC following each sampling event by December 15th of the same year. The reports will describe the sampling event, including any potential deviations from the BMP and will generally follow the report outline suggested in NYSDEC's "Procedures for Collection and Preparation of Aquatic Biota for Contaminant Analysis (Draft)" (2002). The report will include all raw data (including field notes and pictures) and data analyses as described in Section 3.1.3. In addition, summary data will be provided in a table with defined detection limits (NYSDEC, 2002). Where relevant, the biomonitoring report will also make recommendations for any potential modifications to the BMP for the next sampling event. Following the 4th sampling event (5 years post remedy), the biomonitoring report will make a recommendation on the need for further biomonitoring or not, based on a demonstration of a predictable declining trend in PCB tissue concentrations (AECOM, 2021).

[6] Average concentrations will be calculated using half the reporting limit of samples that are not detected.

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SANDERS CREEK
INTERIM CORRECTIVE MEASURE

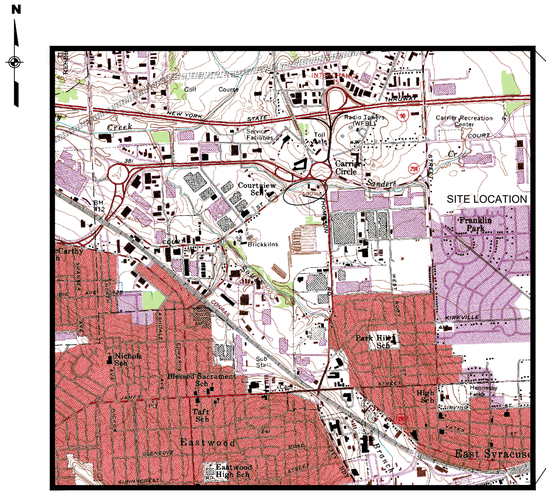
CARRIER SITE

Thompson Road, Syracuse, New York

Corrective Action Order - Index CO 7-20051118-4

NYSDEC Site Registry #734043

SITE PLAN



SITE LOCATION MAP

Prepared by:

AECOM

50 Lakefront Boulevard, Suite 111, Buffalo, New York 14202
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MAY 2023

AECOM

AECOM PROJECT NO. 60626270

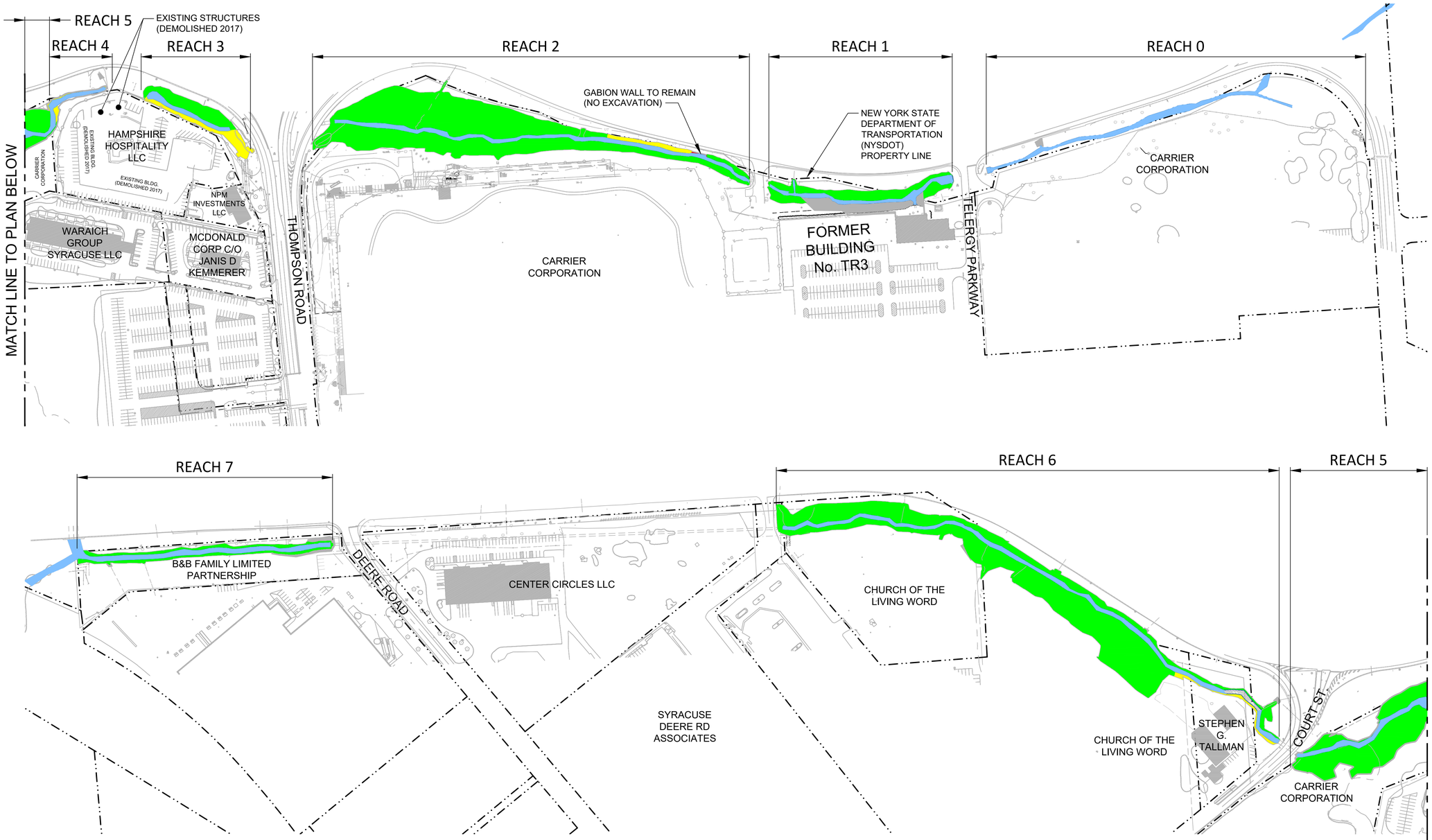
SANDERS CREEK
INTERIM CORRECTIVE MEASURE
CARRIER SITE

Thompson Road, Syracuse, New York

Figure 1.1 Site

Project Management Initials: Designer:____ Checked:____ Approved:____ ANSI D 22" x 34"

Last saved by: HEATHER.PRESSING (2023-05-03) Last Plotted: 2023-05-03
Filename: C:\606262700\00_CAD_GIS\910_CAD\PREV420-SHEETS\FIGURE 1.1.DWG



LEGEND

- APPROXIMATE LIMITS OF SANDERS CREEK / BANK EXCAVATION
- APPROXIMATE PROPERTY BOUNDARY - FROM TAX MAPPING
- PROPOSED AREA OF SOIL REMEDIATION
- PROPOSED AREA OF SOIL REMEDIATION WITH STEEP BANK / INFRASTRUCTURE AREA: ENGINEERED CONTROLS WILL BE IMPLEMENTED IN THE EVENT THAT THE REMEDIATION CRITERIA CANNOT BE ACHIEVED.
- NO EXCAVATION AREA DUE TO SLOPE STABILITY ENGINEERED CONTROL IN-PLACE
- TR-3 PROPOSED AREA OF REMEDIATION TO BE COMPLETED IN CONJUNCTION WITH THIS REMEDIAL ACTION BUT AS PART OF A SEPARATE INTERIM REMEDIAL MEASURE

N

0 150' 300'

1" = 150'

AECOM

PROJECT

SANDERS CREEK INTERIM CORRECTIVE MEASURE

CARRIER SITE
Thompson Road
Syracuse, New York

CLIENT

Carrier Corporation

Thompson Road
Syracuse, New York

CONSULTANT

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PROJECT NUMBER

AECOM #60604770

FIGURE TITLE

Sanders Creek Reaches

FIGURE NUMBER

Figure 1.2

Attachment A

Quality Assurance Project Plan

:

Prepared by:
AECOM
Latham, NY
December 2023

QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR SANDERS CREEK BIOTA MONITORING

CARRIER Corporation Site THOMPSON ROAD, Syracuse, NY

Corrective Action Order – Index CO 7-20051118-4 NYSDEC Site Registry #734043

Prepared for:

Carrier Corporation
Thompson Road
Syracuse, NY 13206

Prepared by:

AECOM
40 British American Blvd
Latham, New York 12110

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1.0 PROJECT DESCRIPTION

1.1 General Overview

The purpose of this Quality Assurance Project Plan (QAPP) is to document planned biota monitoring activities and establish the criteria for completing this monitoring at a predetermined quality for the work conducted by AECOM Technical Services, Inc. (AECOM) for Carrier Corporation (Carrier) under Corrective Action Order – Index CO 7-20051118-4 (CAO). The QAPP is intended to be a companion document to the Biota Monitoring Plan (BMP) and has therefore been included as an appendix in the BMP.

The project description, purpose, and objectives are provided in the BMP. Background data on the site, including the site description and location, site history, previous investigations, and current conditions, are summarized in the Interim Corrective Measures Work Plan (ICMWP) (AECOM, 2021).

Relevant state and federal technical guidance documents that were considered in preparation of the BMP are listed in Section 1 of the BMP. Project work will be conducted in general accordance with the New York State Department of Environmental Conservation (NYSDEC) DER-10, Technical Guidance for Site Investigation and Remediation (NYSDEC, 2010a) and United States Environmental Protection Agency (USEPA) Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA (USEPA, 1988). Per Section 2.4 of DER-10, this QAPP follows the outline in USEPA publication Preparation Aids for the Development of Category I Quality Assurance Project Plans (EPA/600/8-91/003).

1.2 Schedule

Biota monitoring will be completed (to the extent possible) during the same time of year (late summer to early fall) and under similar flow conditions each year. A baseline sampling event will be conducted to document polychlorinated biphenyls (PCB) concentrations in biota tissue prior to the remedial action detailed in the ICMWP. Once the remedial action has been implemented, biota sampling will take place 1, 3, and 5 years post remedy. Data collected during the 4th sampling event (5 years post remedy) will be used to evaluate whether further monitoring is needed.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The general responsibilities of key project personnel for the biota monitoring are listed below. Resumes are provided in Attachment 1.

AECOM Project Manager – Mr. Peter Hollatz P.E. will have responsibility for overall project management and coordination with Carrier and will coordinate the initiation and implementation of the sampling activities.

AECOM Quality Assurance Officer – Ms. Kelly Lurie will serve as the Quality Assurance Officer (QAO) for work completed under the CAO. The QAO will be responsible for oversight of the data validation and laboratory subcontractors, as well as data usability reports. The QAO will work with the AECOM database manager to assure that electronic deliverables provided by the laboratory are accurate and are formatted consistent with AECOM and NYSDEC requirements, as needed.

AECOM Project Chemist – Ms. Ann Kropovitch will serve as the Project Chemist, to be responsible for data validation and preparation of the data usability reports. The Project Chemist will also coordinate with the laboratory regarding analytical questions or concerns, as needed.

Table 2-1 includes contact information for these key personnel, as well as those from the analytical laboratory and for Gradient, a science and engineering consulting firm that is providing technical support related to biota sampling in the context of risk-based remediation. Attachment 1 includes the applicable New York State certifications for the laboratory.

Table 2.1. Contact Information for Key Personnel

Organization	Name	Responsibility	Phone Number	Email
AECOM	Peter Hollatz P.E.	Project Manager	630-918-9648	Peter.Hollatz@aecom.com
AECOM	Kelly Lurie	Quality Assurance Officer	518-542-2944	Kelly.lurie@aecom.com
AECOM	Ann Kropovitch	Project Chemist	-	ann.marie.kropovitch@aecom.com
Gradient	Tim Verslycke, Ph.D.	Project Technical Support	617-395-5594	tverslycke@gradientcorp.com
SGS (Wilmington, NC location)	Tamara Burkamper	Project Manager	910-794-2527	tamara.burkamper@sgs.com
SGS (Wilmington, NC location)	Jeannie Milholland	Quality Assurance	910-616-0140	jeannie.milholland@sgs.com

3.0 QUALITY ASSURANCE OBJECTIVES

Data quality objectives (DQOs) for measurement data in terms of sensitivity and the PARCC parameters (precision, accuracy, representativeness, comparability, and completeness) are established so that the data collected are sufficient and of adequate quality for their intended uses. Data collected and analyzed in conformance with the DQO process described in this QAPP will be used in assessing the uncertainty associated with decisions related to this site. Quality assurance (QA) objectives for precision and accuracy are established in the applicable analytical method and are discussed in detail in the subsections below.

3.1.1 Sensitivity

The sensitivity or detection limit desired is based on the DQOs established for the project. The method detection limit (MDL) is determined in accordance with the procedure in the NYSDEC 2005 Analytical Services Protocol (ASP) Exhibit A, section 4.9.2.12, which is consistent with the procedure in 40 CFR Part 136, Appendix B. The reporting limit (RL) for non-detected PCB aroclors will be a minimum of 0.09 microgram per gram (µg/g). Analytes detected at concentrations below the RL but above the MDL will be flagged "J" (estimated) by the laboratory. The laboratory's reporting limits and MDLs will be reviewed by AECOM's QAO to verify that the laboratory sensitivity is sufficient to meet the project objectives.

3.1.2 Precision

The laboratory objective for precision is to equal or exceed the precision demonstrated for the applied analytical methods on similar samples. Precision is evaluated by the analyses of laboratory and field duplicates, when applicable. Relative Percent Difference (RPD) criteria determined from laboratory performance data are used to evaluate precision between duplicates. Matrix spike/matrix spike duplicate (MS/MSD) analyses will be performed once for every 20 samples.

Precision measures the reproducibility of measurements under a given set of conditions. Specifically, it is a quantitative measure of the variability of a group of measurements compared to their average value. Precision is usually stated in terms of standard deviation, but other estimates such as the coefficient of variation, relative standard deviation, range (maximum value minus minimum value), and relative range are common, and may be used pending review of the data.

The overall precision of measurement data is a mixture of sampling and analytical factors. Analytical precision is easier to control and quantify than sampling precision; there are more historical data related to individual method performance and the "universe" is not limited to the samples received in the laboratory. In contrast, sampling precision is unique to each site or project. Analytical results from laboratory duplicate samples will provide data on measurement (analytical) precision. Field duplicate samples are typically not collected for biota samples since whole bodies are being collected for analysis.

Precision will be determined from matrix spikes and matrix spike duplicates for organic analyses; it will be expressed as the RPD:

$$RPD = 100 \times 2(|X_1 - X_2|) / (X_1 + X_2)$$

where:

X_1 and X_2 are reported concentrations for each duplicate sample and subtracted differences represent absolute values.

Criteria for evaluation of laboratory duplicates are specified in the applicable methods.

3.1.3 Accuracy

The laboratory objective for accuracy is to equal or exceed the accuracy demonstrated for the applied analytical method on similar samples. Percent method recovery criteria and those determined from laboratory performance data are used to evaluate accuracy in matrix (sample) spike and blank spike quality control samples. A matrix spike and blank spike or laboratory control will be performed once for every analytical batch or as specified in the method or ASP. Other method-specific laboratory quality control (QC) samples (such as continuing calibration standards) may also be used in the assessment of analytical accuracy. Sample (matrix) spike recovery is calculated as:

$$\% \text{ Recovery} = 100 \times (SSR-SR)/SA$$

Where:

SSR = Spiked Sample Result;

SR = Sample Result; and

SA = Spike Added

Accuracy measures the bias in a measurement system. It is difficult to measure accuracy for the entire data collection activity. Accuracy will be assessed through use of known QC samples. Accuracy values can be presented in a variety of ways. Accuracy will be normally presented as percent recovery.

Routine organic analytical protocol requires a surrogate spike in each sample. Surrogate recovery will be defined as:

$$\% \text{ Recovery} = (R/S) \times 100$$

Where:

S = surrogate spike concentration

R = reported surrogate compound concentration

Recovery criteria for laboratory spikes and other laboratory QC samples through which accuracy may be evaluated are established in the applicable analytical method.

3.1.4 Representativeness

The representativeness of data is only as good as the representativeness of the samples collected. Sampling and handling procedures, and laboratory practices are designed to provide a standard set of performance-driven criteria to provide data of the same quality as other analyses of similar matrices using the same methods under similar conditions. Representativeness will be determined by a comparison of the quality controls for these samples against data from similar samples analyzed at the same time.

3.1.5 Comparability

Comparability of analytical data among laboratories becomes more accurate and reliable when all labs follow the same procedure and share information for program enhancement. Some of these procedures include:

- Instrument standards traceable to National Institute of Standards and Technology (NIST), the USEPA, or the New York State Department of Health (NYSDOH) or NYSDEC;
- Using standard methodologies;
- Reporting results for similar matrices in consistent units;
- Applying appropriate levels of quality control within the context of the laboratory quality assurance program; and
- Participation in inter-laboratory studies to document laboratory performance.

By using traceable standards and standard methods, the analytical results can be compared to other labs operating similarly. The QA Program documents internal performance. Periodic laboratory proficiency studies are instituted as a means of monitoring intra-laboratory performance. Comparability within any specific project is also assessed by comparison of the project data to data generated previously; and, if available, comparison of the data for multiple sampling events conducted for the project.

3.1.6 Completeness

The goal of completeness is to generate the maximum amount possible of valid data for all planned samples. Completeness of 100 percent indicates that all planned samples were collected; and the resultant data were fully valid and acceptable. As completeness is a function of both field activities and laboratory activities, separate completeness goals are established for each.

The default goal for sampling completeness is 95 percent, as is calculated as

$$\text{Sampling Completeness (\%)} = (Sc/Sp) \times 100$$

Where:

Sc = Samples collected (submitted) for analysis (documented from field records or COC)

Sp = Samples planned (as documented in the BMP or QAPP)

The default goal for analytical completeness is also set at 95 percent. Analytical completeness may be less than 100 percent either due to systemic failures that result in the rejection or loss of data for an entire sample; or compound-specific rejection within an otherwise valid analysis.

For typical projects, the default overall completeness goal is 90 percent useable data. The impact of rejected or unusable data will be made on a case-by-case basis. If the goals of the project can be achieved without the missing datum or data, or if data from a different sampling event can be used to fill the data gap, no further action would be necessary. However, loss of critical data may require resampling or reanalysis.

4.0 SITE SELECTION AND SAMPLING PROCEDURES

Sample locations and sampling procedures, including those for compositing individual biota samples, are discussed in detail in the BMP.

4.1 SAMPLE IDENTIFICATION AND LABELING

Samples will be assigned a unique identification using the species codes listed below.

CR = Crayfish

CC = Creek Chub

WS = White Sucker

LD = Longnose Dase

CF = Catfish

Individual and composite tissue samples will be labeled to include the sampling year, the two letter species code, sampling reach number, and the individual number or composite letter as appropriate (e.g., 24-CR-R0-CompA). Section 2.2.1 of the BMP discusses the nomenclature for individual and composite samples, and Tables 2.3 and 2.4 list the proposed composite sample labels and individual sample IDs, respectively.

Affixed to each sampling container (Ziploc bag) will be a non-removable label on which the following information will be recorded with permanent waterproof ink:

- Site name, location, and job number;
- Sample name;
- Date and time;
- Sampler's name;
- Type of sample (i.e., tissue); and
- Requested analyses.

Field reporting documentation, including field logbooks and field data reporting forms, is discussed in the BMP.

4.2 Sample Preservation

No chemical preservatives are added to biological tissue samples.

4.3 Equipment Decontamination

Prior to moving to a new reach for sampling, all shared materials (e.g., nets, traps, calipers, fish measuring boards, and scales) will be rinsed with creek water to minimize cross contamination between reaches. All used disposable materials (e.g., safety gloves and foil) will be discarded properly.

5.0 SAMPLE CUSTODY

A chain-of-custody form will trace the path of samples from the project site to the laboratory. Chain-of-custody forms are typically provided by the analytical laboratory. The AECOM Project Manager will notify the laboratory of upcoming field sampling events and the subsequent transfer of samples. This notification will include information concerning the number and type of samples, and the anticipated date of arrival. Insulated sample shipping containers (typically coolers) will be provided by the laboratory for shipping samples. Bags containing composite samples within each shipping container will be individually labeled with an adhesive identification label provided by the laboratory.

Once the sample containers (i.e., bags) are filled, they will be immediately placed in the cooler with ice (in Ziploc plastic bags to prevent leaking) or synthetic ice packs to maintain the samples at 4ºC. The field sampler will indicate the sample designation/location number in the space provided on the chain-of-custody form for each sample. The chain-of-custody forms will be signed and placed in a sealed plastic Ziploc bag in the cooler. The completed shipping container will be closed for transport with nylon strapping, or a similar shipping tape, and two paper seals will be affixed to the lid. The seals must be broken to open the cooler and will indicate tampering if the seals are broken before receipt at the laboratory. A label may be affixed identifying the cooler as containing “Environmental Samples” and the cooler will be shipped by an overnight delivery service to the laboratory. When the laboratory receives the coolers, the custody seals will be checked and lab personnel will sign the chain-of-custody form.

5.1 LABORATORY SAMPLE RECEIPT

Upon receipt at the laboratory, a laboratory representative inspects the samples for integrity and checks the shipment against the chain-of-custody. Discrepancies are addressed at this point and documented on the chain-of-custody form and the cooler checklist. Discrepancies are reported to the Laboratory Project Manager who contacts the AECOM Project Manager or QAO for resolution.

When the shipment and the chain-of-custody are in agreement, the custodian enters the samples into the Laboratory Information Management System and assigns each sample a unique laboratory number. This number is affixed to each sample container. The custodian then enters the sample and analysis information into the laboratory computer system.

5.1.1 Laboratory Sample Custody

The laboratory must satisfy the sample chain-of-custody requirements by implementing the following procedures for laboratory/sample security:

- Samples are stored in a secure area;
- Access to the laboratory is through a monitored area;
- Visitors sign a visitor’s log and are escorted while in the laboratory;
- Only the designated sample custodians have keys to sample storage area(s); and
- Transfers of samples in and out of storage are documented.

5.1.2 Sample Storage, Security, and Disposal

While in the laboratory, the samples and aliquots that require storage at 4°C ± 2°C are maintained in a locked refrigerator unless they are being used for analysis. The laboratory is responsible for sample storage and security so that:

- Samples and extracts are stored for 60 days after the final analytical data report has been submitted to AECOM. The samples, extracts, and digestates are then disposed by the laboratory in accordance with laboratory standard operating procedures (SOPs) and applicable regulations.
- Samples are not stored with standards or sample extracts

6.0 CALIBRATION PROCEDURES AND FREQUENCY

Quality assurance for instrumentation and equipment used for a project is controlled by a formal calibration program, which verifies that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements. Instruments and equipment that measure a quantity, or whose performance is expected at a stated level, are subject to calibration. Calibration is performed using reference standards or externally by calibration agencies or equipment manufacturers.

6.1 FIELD EQUIPMENT CALIBRATION

The water quality meter used in the field during sample collection to measure water quality parameters at the time and location of sample collection will be calibrated daily following manufacturer's instructions. An example of the Daily Calibration Form to be used is provided in Attachment 2.

6.2 LABORATORY EQUIPMENT CALIBRATION

Laboratory equipment will be calibrated according to the method-specific requirements of the 2005 NYSDEC ASP**,** Exhibit E, Parts II and III, and maintained following professional judgment and the manufacturer's specifications, and additional requirements as specified in the Environmental Laboratory Accreditation Program (ELAP) certification manual.

6.2.1 Calibration Procedure

Written procedures are used for all instruments and equipment subject to calibration. For chemical analyses typically performed for this contract, the calibration procedures are specified in the methods as compiled in the ASP. If established procedures are not available, a procedure is developed considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operational error on the quantities measured.

6.2.2 Calibration Frequency

Calibration frequency is based on the type of equipment, inherent stability, manufacturer's recommendations, values provided in recognized standards, intended data use, specified analytical methods, effect of error upon the measurement process, and prior experience.

6.2.3 Calibration Reference Standards

Two types of reference standards will be used by the standby laboratories for calibration:

Physical standards, such as weights for calibrating balances and certified thermometers for calibrating working thermometers, refrigerators and ovens, are generally used for periodic calibration.

Chemical standards, such as Standard Reference Materials (SRMs) provided by the NIST or USEPA, may also include vendor-certified materials traceable to NIST or USEPA SRMs. These are primarily used for operational calibration.

6.2.4 Calibration Failure

Equipment that cannot be calibrated or becomes inoperable is removed from service. Such equipment must be repaired and satisfactorily recalibrated before re-use. For laboratory equipment that fails calibration, analysis cannot proceed until appropriate corrective action is taken and the analyst achieves an acceptable calibration.

Laboratory managers are responsible for development and implementation of a contingency plan for major equipment failure. The plan includes guidelines on waiting for repairs, use of other instrumentation, subcontracting analyses, and evaluating scheduled priorities.

6.2.5 Calibration Records

Records are prepared and maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of preparation, stability, and proof of continuity of reference standards are also maintained. Copies of the raw calibration data are kept with the analytical sample data.

6.3 OPERATIONAL CALIBRATION

Operational calibration is generally performed as part of the analytical procedure and refers to those operations in which instrument response (in its broadest interpretation) is related to analyte concentration. Included are the preparation of a standard response (calibration) curve and often the analysis of blanks.

Preparation of a standard calibration curve is accomplished by the analysis of calibration standards, which are prepared by adding the analyte(s) of interest to the solvent that is introduced into the instrument. The concentrations of the calibration standards are chosen to cover the working range of the instrument or method. For most methods, five calibration standards are used, with the concentration of the lowest calibration standard being the reporting or quantitation limit for that analysis. Sample measurements are made and reported within this working range; apparent concentrations which exceed the high end of the calibrated range (“E”-flagged data for organic analyses) are diluted (or a smaller sample is used) and re-analyzed. The calibration curve is prepared by plotting or performing a linear regression (or quadratic where necessary) of the instrument responses against the analyte concentration.

7.0 ANALYTICAL PROCEDURES AND CALIBRATION

7.1 ANALYTICAL METHODS

Sample analyses for this biota monitoring program will utilize USEPA SW-846 methods as listed below.

- PCBs – SW 846 Method 8082A
- Percent Moisture – Method 3540C or 3545C
- Lipids

Analytical methods used for this CAO are presented in the NYSDEC ASP, 2005. It is the laboratory's responsibility to be familiar with this document and procedures and deliverables within it pertaining to New York State work. Full Category B deliverables will be required.

AECOM may use SGS North America, Inc. (SGS) for this project. SGS is certified by the NYSDOH ELAP and is in good standing for the applicable parameter groups. SGS' NYSDOH certification for the applicable methods is provided in Attachment 1, and the applicable SOPs are provided in Attachment 3.

8.0 DATA REDUCTION, VALIDATION, AND REPORTING

The guidance followed to perform quality data validation, and the methods and procedures outlined herein pertain to initiating and performing data validation, as well as reviewing data validation performed by others (if applicable). An outline of the data validation process is presented here, followed by a description of data validation review summaries.

8.1 LABORATORY DATA REPORTING AND REDUCTION

Data reduction is the process by which raw analytical data generated from laboratory instrument systems is converted into usable concentrations. The raw data, which may take the form of area counts, instrument responses, or observations, are processed by the laboratory and converted into concentrations expressed in the parts per million (mg/kg or mg/L) or parts per billion (µg/kg or µg/L) range. Raw data from these systems include compound identifications, concentrations, retention times, and data system print-outs. Raw data are usually reported in graphic form, bar graph form, or tabular form. The laboratory will follow standard operating procedures consistent with the data handling requirements of the applicable methods.

The laboratory will meet the applicable documentation, data reduction, and reporting protocols as specified in the 2005 revision of the NYSDEC ASP. ASP Deliverables will be Category B (full or CLP-equivalent deliverables). Laboratory data reports will conform to NYSDEC Category B deliverable requirements, as specified in Exhibit B, Part II.E, Sections 2 and 3, respectively.

Copies of the laboratory's generic Quality Assurance Management Plan (QAMP, as defined in ASP 2005 Exhibit E, Part I) will be maintained at AECOM's office (Latham, NY). The laboratory's QAMP will indicate the standard methods and practices for obtaining and assessing data, and how data are reduced from the analytical instruments to a finished report, indicating levels of review along the way.

To meet NYSDEC electronic data deliverable (EDD) requirements, the laboratory for this work will be required to submit electronic deliverables in an EQuIS 4-file format consistent with AECOM standards (see Attachment 4). AECOM's database manager will be responsible verifying that the file submitted meets these specifications including verifying that current NYSDEC Valid Values were used for sample coding; providing an Excel (or Access) file to the data validator; uploading the validated data into the database; overseeing the uploading of any other data (field data, etc.), and submitting a final EQuIS deliverable to NYSDEC that meets NYSDEC EDD requirements.

In addition to the hard copy of the data report, the laboratory will be asked to provide the sample data in spreadsheet form (submitted electronically or on computer diskette). The data spreadsheet will be generated to the extent possible directly from the laboratory's electronic files or information management system to minimize possible transcription errors resulting from the manual transcription of data.

8.2 DATA VALIDATION

Data generated for projects under this contract will be validated by an AECOM chemist. The validator will follow guidelines established in the USEPA Region 2 SOPs applicable to the analytical method(s) being reviewed. These SOPs are checklists which are designed to formally and rigorously assess the quality and completeness of SW-846 analysis data packages. The use of these USEPA SOPs will be adapted to conform to the specific requirements of the NYSDEC ASP (e.g., NYSDEC/ASP holding times; matrix spike blank requirements). Where necessary and appropriate, supplemental validation criteria may be derived from the EPA Functional Guidelines (USEPA Contract Laboratory Program

National Functional Guidelines for Inorganic Data Review, EPA-540-R-013-001, August 2014, and the National Functional Guidelines for Organic Data Review, EPA-540-R-014-002, August 2014).

Validation reports and data usability summary reports (DUSR) will consist of text results of the review and marked up copies of Form I (results with qualifiers applied by the validator). Results with validation qualifiers may also be added to the Excel EDD data file provided by the lab. Validation will consist of target and non-target compounds with corresponding method blank data, spike and surrogate recoveries, sample data, and a final note of validation decision or qualification, along with any pertinent footnote references. Qualifiers applied to the data will be documented in the report text. Where QC failures caused the laboratory to perform a re-analysis, the data validator will make a recommendation as to which of the two analyses should be used. Data review will also include an assessment of sensitivity (i.e., are reporting limits appropriate to determine if contaminants are present at or above action levels or other applicable threshold values).

8.3 DATA USABILITY

Subsequent to review of the items evaluated in the data validation reports and accompanying tables, AECOM's chemist will prepare a DUSR which encompasses both quantitative and qualitative aspects, although the qualitative element is the most significant.

The quantitative aspect is a summary of the data quality as expressed by qualifiers applied to the data; the percent rejected, qualified (i.e., estimated), missing, and fully acceptable data are reported. As appropriate, this quantitative summary is broken down by matrix, laboratory, or analytical fraction or method.

The qualitative element of the DUSR is the translation and summary of the validation reports into a discussion useful to data users. The qualitative aspect will discuss the significance of the qualifications applied to the data, especially in terms of those most relevant to the intended use of the data. The usability report will also indicate whether there is a suspected bias (high or low) in qualified data, and will also provide a subjective overall assessment of the data quality. If similar analyses are performed by more than one method, a discussion of the extent of agreement among the various methods will be included, as well as discussion of any discrepancies among the data sets.

The DUSR will indicate if there is a technical basis for selecting one data type over another for multiple measurements which are not in agreement.

Data which has not been validated and field data used for the project will be discussed in the data usability summary, including any limitations on the use of such data.

8.4 DATA REPORTING

Reporting for the biota monitoring is detailed in the BMP. The final report summarizing the data will include a QA section that documents QA/QC activities that were completed as well as the results and any limitations of the data in meeting the QA objectives.

9.0 INTERNAL QUALITY CONTROL CHECKS

9.1 FIELD QUALITY ASSURANCE

Field personnel will record all field data in bound field logbooks and on standard forms. After checking the validity of the data in the field notes, the AECOM Project Manager or his/her designee will reduce the data to tabular form, when possible, by entering the data into data files. Where appropriate, the data files will be set up for direct input into the project database. Subjective data will be filed as hard copies for later review by the Project Manager and incorporation into technical reports, as appropriate.

Verification of field data will be performed at two different levels. The first level of data verification will be performed at the time of collection by following standard procedures and QC checks. The second level of review consists of the Project Manager, Task Manager, or other competent personnel, reviewing the data to confirm that the correct codes and units have been included. After data reduction into tables and arrays is complete, the Site Investigation Lead will review data sets for anomalous values. The Project Manager, who will review field reports for reasonableness and completeness, will validate subjective field and technical data.

9.2 LABORATORY QUALITY ASSURANCE

9.2.1 Method Blanks

A method blank is laboratory water on which every step of the method is performed and analyzed along with the samples. Method blanks are used to assess the background variability of the method and to assess the introduction of contamination to the samples by the method, technique, or instruments as the sample is prepared and analyzed in the laboratory. Method blanks will be analyzed at a frequency of one for every twenty samples analyzed or as otherwise specified in the analytical protocol.

9.2.2 Laboratory Duplicates

Laboratory duplicates are sub-samples taken from a single aliquot of sample after the sample has been thoroughly mixed or homogenized (with the exception of volatile organics), to assess the precision or reproducibility of the analytical method on a sample of a particular matrix.

9.2.3 Spiked Samples

Two types of spiked samples will be prepared and analyzed as quality controls: matrix spikes and matrix spike duplicates, which are analyzed to evaluate instrument and method performance and performance on samples of similar matrix. MS/MSD samples will be analyzed at a frequency of one (pair) for every 20 samples. In addition, matrix spike blanks (MSBs) will also be prepared and analyzed by the laboratory as required by NYSDEC ASP.

9.2.4 Laboratory Control Sample

A fortified clean matrix (laboratory control sample, or LCS) is analyzed with each analysis. In some cases a “Laboratory-Fortified Blank” (LFB) may serve as the LCS. These samples generally consist of a standard aqueous or solid matrix fortified with the analytes of interest for single-analyte methods and selected analytes for multi-analyte methods according to the appropriate analytical method. The LCS may be analyzed in duplicate for some methods (LCSD). The analyte recovery from each analysis (LCS and LCSD) is used to monitor analytical accuracy; analytical precision can be assessed from evaluation of the LCS/LCSD in the same manner as the MS/MSD.

10.0 PERFORMANCE AND SYSTEM AUDITS

Audits are systematic checks to determine the quality of operation of some activity or function in the field or laboratory. Field audits are conducted to verify adherence to proper field and sampling procedures. Audits are of two types, as described below:

- Performance audits are independent safety and health, procedure, and/or sample checks made by a supervisor or auditor to arrive at a quantitative measure of the quality of the data produced by one section or the entire measurement process.
- System audits are onsite qualitative inspections and reviews of the QA system used by some part of or the entire measurement system. The audits are performed against the QAPP. A checklist is typically generated from the requirements and becomes the basis for the audit. The results of any deficiencies noted during the audit are summarized in an audit report.

Laboratory performance and system audits are performed by the laboratory's QA staff to assess the effectiveness of the quality system. These internal audits are performed on a routine basis. Audits are also performed by certifying agencies. Audit reports and corrective actions are available to NYSDEC for review.

10.1 RESPONSIBILITY, AUTHORITY, AND TIMING

QA audits that may be conducted for the project include system, performance, and data audits. The Project QA Officer will keep records for all completed audits in the project files.

10.2 FIELD AUDITS

The need for field audits will be determined on an ongoing basis. Not all the aspects listed below may be necessary or appropriate for all circumstances.

Field performance audits, if specified, will be conducted during the project as field data are generated, reduced, and analyzed. Numerical manipulations, including manual calculations, will be documented. Records of numerical analyses will be legible, of reproduction quality, and sufficiently complete to permit logical reconstruction by a qualified individual other than the originator.

System audits of site activities will be accomplished by an inspection of all field site activities. During this audit, the auditor(s) will compare current field practices with proposed procedures. The following elements will be evaluated during a field system audit:

- Field activities conducted in substantial compliance with the BMP
- Procedures and analyses conducted according to procedures outlined in the QAPP
- Sample documentation
- Working order of instruments and equipment
- Level of QA conducted by field personnel
- Contingency plans in case of equipment failure or other event preventing the planned activity from proceeding
- Decontamination procedures

- Level of efficiency with which each team conducts planned activities at one site and proceeds to the next
- Sample packaging and shipment.

After completion of the audit, any deficiencies will be discussed with the field staff and corrections identified. If any of these deficiencies could affect the integrity of the samples being collected, the auditor(s) will inform the field staff and corrections will be implemented immediately. The audit will be performed by the Project QA/QC Coordinator or designee.

10.3 LABORATORY PERFORMANCE AND SYSTEM AUDITS

The laboratory assigned to this project will be verified to be certified by the NYSDOH ELAP for the matrices and analytical protocols to be used. Therefore, no project-specific audit of the laboratory(s) will be performed unless warranted by a problem(s) that cannot be resolved by any other means.

10.4 AUDIT PROCEDURES

Prior to an audit, the designated lead auditor prepares an audit checklist. During an audit and upon its completion, the auditor(s) will discuss the findings with the individuals audited and discuss and agree on corrective actions to be initiated. The auditor will then prepare and submit an audit report to the manager of the audited group and the Project Manager.

The manager of the audited group will then prepare and submit, to the Project QA Officer and the Project Manager, a plan for implementing the corrective action to be taken on non-conformances indicated in the audit report, the date by which such corrective action will be completed, and actions taken to prevent reoccurrence. If the corrective action has been completed, supporting documentation should be attached to the reply. The auditor will ascertain (by re-audit or other means) if appropriate and timely corrective action has been implemented.

Records of audits will be maintained in the project files.

10.5 AUDIT DOCUMENTATION

A checklist will be completed during each audit so that the previously defined scope of the individual audits is accomplished and that the audits follow established procedures. The checklist will detail the activities to be executed as part of the auditing plan. Audit checklists will be prepared in advance and will be available for review. Following each system, performance, and data audit, the auditor or QAO will prepare a report to document the findings of the specific audit.

11.0 PREVENTIVE MAINTENANCE

Preventive maintenance is not required for the scope of work discussed in the BMP.

12.0 CALCULATION OF DATA QUALITY INDICATORS

Equations to be used for precision, accuracy, and completeness are provided above in Section 3. Method detection limits will be specified in the applicable methods, and the reporting limit RL for non-detected PCB aroclors will be 0.09 µg/g.

13.0 CORRECTIVE ACTION

If instrument performance or data fall outside acceptable limits, then corrective actions will be taken. These actions may include recalibration or standardization of instruments, acquiring new standards, replacing equipment, repairing equipment, and reanalyzing samples or redoing sections of work.

Subcontractors providing analytical services should perform their own internal laboratory audits and calibration procedures with data review conducted at a frequency so that errors and problems are detected early, thus avoiding the prospect of redoing large segments of work.

Situations related to this project requiring corrective action will be documented and made part of the project file. For each measurement system identified requiring corrective action, the responsible individual for initiating the corrective action and also the individual responsible for approving the corrective action, if necessary, will be identified.

As part of its quality management system (QMS) program, AECOM provides relevant excerpts and conclusions from data validation reports to the analytical laboratories. The laboratories are therefore made aware of non-critical items and areas where improvement may be made.

The objectives of the corrective action procedures presented below are to ensure that recognized errors in performance of sample and data acquisition lead to effective remedial measures and that those steps are documented to provide assurance that any data quality deficiencies are recognized in later interpretation and are not recurrent.

13.1 RATIONALE

Many times corrective measures are undertaken in a timely and effective fashion but go undocumented. In other cases, corrective actions are of a complex nature and may require scheduled interactions between departmental groups. In either case, documentation in a formal or informal sense can reinforce the effectiveness and duration of the corrective measures taken.

13.2 CORRECTIVE ACTION METHODS

13.2.1 Immediate Corrective Actions

Immediate corrective actions are of a minor or routine nature such as correcting malfunctioning equipment, correction of data transcription errors, and other such activities routinely made in the field, laboratory, or office by technicians, analysts, and other project staff.

13.2.2 Long-Term Corrective Actions

Long-term corrective action will be used to identify and eliminate causes of non-conformances which are of a complex nature and that are formally reported between management groups.

13.2.3 Corrective Action Steps

For long-term corrective actions, steps comprising closed-loop corrective action system are as follows:

- Define the problem;
- Assign responsibility for investigating the problem;

- Investigate and determine the cause of the problem;
- Determine a corrective action to eliminate the problem;
- Assign and accept responsibility for implementing the corrective action; and
- Verify that the corrective action has eliminated the problem.

Non-conformance events associated with analytical work are documented by the laboratory's Non-Conformance Records, which are reviewed and approved by the laboratory's Quality Assurance Manager.

13.2.4 Audit-Based Non-Conformances

Following audits, corrective action is initiated by documenting the audit finding and recommended corrective action on an Audit Finding Report.

13.3 CORRECTIVE ACTION REPORT REVIEW AND FILING

Immediate and long-term corrective actions require review to assure that, during the time of non-conformance, erroneous data were not generated or that, if possible, correct data were acquired instead. Such confirmation and review is the responsibility of the supervisor of the staff implementing the corrective action. Confirmation will be acknowledged by notation and dated signature on the affected data record or appropriate form or by memorandum to AECOM project management.

14.0 QUALITY CONTROL REPORTS TO MANAGEMENT

Fundamental to the success of this QA/QC is the active participation of the AECOM Project Manager and the Project QA Officer. The Project QA Officer will be advised of project activities and will participate in development, review, and operation of the project. Project management will be informed of QA activities through the receipt, review, and/or approval of:

- Project-specific work plans;
- Corrective action notices; and
- Non-conformance records.

The Project Manager, through task managers, will be responsible for verifying that records and files related to the project are stored appropriately and are retrievable.

The laboratory will submit any memoranda or correspondence related to quality control of this project's samples as part of its deliverables package.

15.0 REFERENCES

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USEPA, 1986. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, Third edition. EPA SW-846. With revisions and updates through March, 2009. Accessed on line (at “SW-846 On-Line”) May 2011 at http://www.epa.gov/epaoswer/hazwaste/test/main.htm

USEPA, 1988. *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA*. USEPA Office of Emergency and Remedial Response. OSWER Directive No. 355.3-01. October.

Attachments

Attachment 1

Lab Credentials and Resumes for Key Project Personnel

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2024
Issued April 01, 2023

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. GREG DICKINSON
SGS NORTH AMERICA INC. - WILMINGTON
5500 BUSINESS DRIVE
WILMINGTON, NC 28405

NY Lab Id No: 11685

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Dioxins and Furans

1,2,3,4,6,7,8,9-Octachlorodibenzofura	EPA 8290A
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-d	EPA 8290A
1,2,3,4,6,7,8-Heptachlorodibenzofurar	EPA 8290A
1,2,3,4,6,7,8-Heptachlorodibenzo-p-di	EPA 8290A
1,2,3,4,7,8,9-Heptachlorodibenzofurar	EPA 8290A
1,2,3,4,7,8-Hexachlorodibenzofuran	EPA 8290A
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,6,7,8-Hexachlorodibenzofuran	EPA 8290A
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,7,8,9-Hexachlorodibenzofuran	EPA 8290A
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,7,8-Pentachlorodibenzofuran	EPA 8290A
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	EPA 8290A
2,3,4,6,7,8-Hexachlorodibenzofuran	EPA 8290A
2,3,4,7,8-Pentachlorodibenzofuran	EPA 8290A
2,3,7,8-Tetrachlorodibenzofuran	EPA 8290A
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290A

Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
PCB 1	EPA 1668A
PCB 10	EPA 1668A

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Polychlorinated Biphenyls

PCB 100	EPA 1668A
PCB 101	EPA 1668A
PCB 102	EPA 1668A
PCB 103	EPA 1668A
PCB 104	EPA 1668A
PCB 105	EPA 1668A
PCB 106	EPA 1668A
PCB 107	EPA 1668A
PCB 108	EPA 1668A
PCB 109	EPA 1668A
PCB 11	EPA 1668A
PCB 110	EPA 1668A
PCB 111	EPA 1668A
PCB 112	EPA 1668A
PCB 113	EPA 1668A
PCB 114	EPA 1668A
PCB 115	EPA 1668A
PCB 116	EPA 1668A
PCB 117	EPA 1668A
PCB 118	EPA 1668A
PCB 119	EPA 1668A
PCB 12	EPA 1668A
PCB 120	EPA 1668A
PCB 121	EPA 1668A
PCB 122	EPA 1668A
PCB 123	EPA 1668A
PCB 124	EPA 1668A

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Polychlorinated Biphenyls

PCB 125	EPA 1668A
PCB 126	EPA 1668A
PCB 127	EPA 1668A
PCB 128	EPA 1668A
PCB 129	EPA 1668A
PCB 13	EPA 1668A
PCB 130	EPA 1668A
PCB 131	EPA 1668A
PCB 132	EPA 1668A
PCB 133	EPA 1668A
PCB 134	EPA 1668A
PCB 135	EPA 1668A
PCB 136	EPA 1668A
PCB 137	EPA 1668A
PCB 138	EPA 1668A
PCB 139	EPA 1668A
PCB 14	EPA 1668A
PCB 140	EPA 1668A
PCB 141	EPA 1668A
PCB 142	EPA 1668A
PCB 143	EPA 1668A
PCB 144	EPA 1668A
PCB 145	EPA 1668A
PCB 146	EPA 1668A
PCB 147	EPA 1668A
PCB 148	EPA 1668A
PCB 149	EPA 1668A

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Polychlorinated Biphenyls

PCB 15	EPA 1668A
PCB 150	EPA 1668A
PCB 151	EPA 1668A
PCB 152	EPA 1668A
PCB 153	EPA 1668A
PCB 154	EPA 1668A
PCB 155	EPA 1668A
PCB 156	EPA 1668A
PCB 157	EPA 1668A
PCB 158	EPA 1668A
PCB 159	EPA 1668A
PCB 16	EPA 1668A
PCB 160	EPA 1668A
PCB 161	EPA 1668A
PCB 162	EPA 1668A
PCB 163	EPA 1668A
PCB 164	EPA 1668A
PCB 165	EPA 1668A
PCB 166	EPA 1668A
PCB 167	EPA 1668A
PCB 168	EPA 1668A
PCB 169	EPA 1668A
PCB 17	EPA 1668A
PCB 170	EPA 1668A
PCB 171	EPA 1668A
PCB 172	EPA 1668A
PCB 173	EPA 1668A

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Polychlorinated Biphenyls

PCB 174	EPA 1668A
PCB 175	EPA 1668A
PCB 176	EPA 1668A
PCB 177	EPA 1668A
PCB 178	EPA 1668A
PCB 179	EPA 1668A
PCB 18	EPA 1668A
PCB 180	EPA 1668A
PCB 181	EPA 1668A
PCB 182	EPA 1668A
PCB 183	EPA 1668A
PCB 184	EPA 1668A
PCB 185	EPA 1668A
PCB 186	EPA 1668A
PCB 187	EPA 1668A
PCB 188	EPA 1668A
PCB 189	EPA 1668A
PCB 19	EPA 1668A
PCB 190	EPA 1668A
PCB 191	EPA 1668A
PCB 192	EPA 1668A
PCB 193	EPA 1668A
PCB 194	EPA 1668A
PCB 195	EPA 1668A
PCB 196	EPA 1668A
PCB 197	EPA 1668A
PCB 198	EPA 1668A

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Polychlorinated Biphenyls

PCB 199	EPA 1668A
PCB 2	EPA 1668A
PCB 20	EPA 1668A
PCB 200	EPA 1668A
PCB 201	EPA 1668A
PCB 202	EPA 1668A
PCB 203	EPA 1668A
PCB 204	EPA 1668A
PCB 205	EPA 1668A
PCB 206	EPA 1668A
PCB 207	EPA 1668A
PCB 208	EPA 1668A
PCB 209	EPA 1668A
PCB 21	EPA 1668A
PCB 22	EPA 1668A
PCB 23	EPA 1668A
PCB 24	EPA 1668A
PCB 25	EPA 1668A
PCB 26	EPA 1668A
PCB 27	EPA 1668A
PCB 28	EPA 1668A
PCB 29	EPA 1668A
PCB 3	EPA 1668A
PCB 30	EPA 1668A
PCB 31	EPA 1668A
PCB 32	EPA 1668A
PCB 33	EPA 1668A

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Polychlorinated Biphenyls

PCB 34	EPA 1668A
PCB 35	EPA 1668A
PCB 36	EPA 1668A
PCB 37	EPA 1668A
PCB 38	EPA 1668A
PCB 39	EPA 1668A
PCB 4	EPA 1668A
PCB 40	EPA 1668A
PCB 41	EPA 1668A
PCB 42	EPA 1668A
PCB 43	EPA 1668A
PCB 44	EPA 1668A
PCB 45	EPA 1668A
PCB 46	EPA 1668A
PCB 47	EPA 1668A
PCB 48	EPA 1668A
PCB 49	EPA 1668A
PCB 5	EPA 1668A
PCB 50	EPA 1668A
PCB 51	EPA 1668A
PCB 52	EPA 1668A
PCB 53	EPA 1668A
PCB 54	EPA 1668A
PCB 55	EPA 1668A
PCB 56	EPA 1668A
PCB 57	EPA 1668A
PCB 58	EPA 1668A

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All approved analytes are listed below:

Polychlorinated Biphenyls

PCB 59	EPA 1668A
PCB 6	EPA 1668A
PCB 60	EPA 1668A
PCB 61	EPA 1668A
PCB 62	EPA 1668A
PCB 63	EPA 1668A
PCB 64	EPA 1668A
PCB 65	EPA 1668A
PCB 66	EPA 1668A
PCB 67	EPA 1668A
PCB 68	EPA 1668A
PCB 69	EPA 1668A
PCB 7	EPA 1668A
PCB 70	EPA 1668A
PCB 71	EPA 1668A
PCB 72	EPA 1668A
PCB 73	EPA 1668A
PCB 74	EPA 1668A
PCB 75	EPA 1668A
PCB 76	EPA 1668A
PCB 77	EPA 1668A
PCB 78	EPA 1668A
PCB 79	EPA 1668A
PCB 8	EPA 1668A
PCB 80	EPA 1668A
PCB 81	EPA 1668A
PCB 82	EPA 1668A

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Polychlorinated Biphenyls

PCB 83	EPA 1668A
PCB 84	EPA 1668A
PCB 85	EPA 1668A
PCB 86	EPA 1668A
PCB 87	EPA 1668A
PCB 88	EPA 1668A
PCB 89	EPA 1668A
PCB 9	EPA 1668A
PCB 90	EPA 1668A
PCB 91	EPA 1668A
PCB 92	EPA 1668A
PCB 93	EPA 1668A
PCB 94	EPA 1668A
PCB 95	EPA 1668A
PCB 96	EPA 1668A
PCB 97	EPA 1668A
PCB 98	EPA 1668A
PCB 99	EPA 1668A

Serial No.: 67118

Ann Marie Kropovitch

Environmental Chemist

Education
BA, Chemistry
State University College of New York, Buffalo, NY,1987

Years of experience
36 total | 23 with AECOM

Training
OSHA 40-hour HAZWOPER

Summary

Ms. Kropovitch is an Environmental Chemist with hands-on experience at commercial environmental laboratories and a hazardous waste disposal facility. She is currently involved in field sampling of soil/groundwater, laboratory coordination, data validation (for both internal and external clients of AECOM) and data usability for compliance monitoring and remedial investigations. Responsibilities at past employers have included laboratory analysis, internal Laboratory Information Management System (LIMS) support, review of WQ and metals data, Project Manager and Supervisory functions (workload scheduling, interviewing/hiring of employees, progress and confidential meetings with management).

Project Experience

EPA, HI - provided validation support for a time critical project. Also involved in tracking and verifying invoices submitted from the laboratories.

McConnel Air Force Base – providing validation support for AECOM Omaho office as needed.

Plattsburgh Air Force Base, New York: Responsible for all communication between the on-site office and analytical laboratories. This includes obtaining sampling containers, laboratory scheduling, and problem resolution. Validation of all laboratory reports for the site in accordance with the AFCEE QAPP 4.0. Responsible for the generation of data assessment reports and updating/maintenance of the database (ACCESS). Familiar with the use of ACCESS/EXCEL/WORD.

New York State Department of Environmental Conservation (NYSDEC) Various Projects: (some examples are 315 N. Meadow Street, College Point, E. 90th Street, Home Depot, Kleigman Brothers, Meeker Avenue, N. of 720 Melrose Avenue, 315 N. Meadow Street, Oser Avenue, Ozone Industries, Polymer Applications site, Tonawanda Forge, and West Side): Responsible for communication between the on-site samplers and the analytical laboratory. This included obtaining sampling containers, coordinating laboratory availability, and problem resolution. Responsible for updating/maintenance of the databases. Reviewed laboratory reports and prepared Data Usability Summary Reports (DUSR) in accordance with NYSDEC guidelines.

Orphan Mine Soil Investigation, National Parks Service (2012): Provided validation support for the URS Denver office - reviewed organics, metals, radiological and wet chemistry parameters in order to meet a time critical deadline. Also involved in report generation and table construction.

US Steel (2009-2012): Provided validation support for the URS Chicago office in the review of organics, metals, and wet chemistry parameters. Also involved in report generation and table construction.

Pfohl Brothers Landfill, Town of Cheektowaga, New York (2002-current): Performed groundwater sampling as part of the on-site team. Also responsible for communication between the on-site samplers and the analytical laboratory. This includes obtaining sampling containers, coordinating laboratory availability, and problem resolution. Responsible for updating/maintenance of the database. Reviewed laboratory reports and prepared Data Assessment Reports (DAR) in accordance with NYSDEC guidelines.

US Army Corps of Engineers Projects: Baltimore District projects including Chillum Spill Site and Mattioni-American Recovery. Responsible for validation of analytical data and creation of validation reports in accordance with USEPA guidelines.

- **Bailey Creek, DOL Storage Yard (Fort Eustis, Virginia), Felker Fuel Farm (Fort Eustis, Virginia) - Malcolm Pirnie:** Performed data validation in accordance with the USEPA Region III guidelines for various organic and inorganic parameters. Clarified potential data problems with the laboratory and prepared validation reports in accordance with USEPA guidelines.

- **Coopers Creek, Boone Creek, Cluster 19, and Hogs Point at the Aberdeen Proving Ground - General Physics:** Performed data validation in accordance with the USEPA Region III guidelines for various organic and inorganic parameters including

explosives. Clarified potential data problems with the laboratory and prepared validation reports in accordance with USEPA guidelines.

- **Consolidated Edison Company - Former MPG (Manufactured Gas Plant) site:** Performed data validation in accordance with the USEPA Region II guidelines for various organic and inorganic parameters. Clarified potential data problems with the laboratory. Prepared DUSRs in accordance with NYSDEC guidelines.

Titan Missile Site: Responsible for validation of analytical data and preparation of a Quality Control Summary Report (QCSR) and Analytical Data Packages (ADP).

Laboratory Analysis:
Metals - Used atomic absorption (AA) and inductively coupled plasma (ICP) to analyze non-potable water, soil, and waste samples.

Water Quality - Familiar with various techniques including filtration, distillation, colorimetric methods, and incubation. Actual analysis performed included: total organic carbon, phenols, biochemical oxygen demand, cyanide, fecal coliform, chemical oxygen demand, and ammonia.

PCBs - Used an HP5890 gas chromatograph to analyze both soil and oil hazardous waste samples for PCBs. Familiar with the extraction of water samples using separatory funnels and soils using the soxhlet technique.

Data Review: Performed quality control (QC) review on metals and WQ data at a commercial laboratory as the supervisor of the inorganic review group. Familiar with both LIMS data and contract laboratory protocol (CLP) metals forms and QC requirements. Used both the Ward® and Metals Analytical Reporting System (MARS®) software for the processing of metals following USEPA and NYSDEC guidelines.

Validation Review: Validated reports as a 3rd party (outside clients) and in-house (URS Buffalo/Chicago/Denver offices). Using guidelines provided by the USEPA and in accordance with various methods I review for usability Volatiles, Semivolatile, Pesticide/PCB, Metals, Wet Chemistry, and Radiochemistry data.

Project Manager: Interacted with several clients as a Project Manager at a commercial laboratory. Responsible for all client contact and problem resolution. Created bottle orders, verified chain-of-custody (COC) when samples arrived, kept clients informed of problems or delays, and approved final reports.

Supervision: Past experience includes supervision of an inorganic processing group and metals laboratory. Handled day-to-day operational issues such as workload scheduling and supply ordering. Also conducted training and maintenance of documents pertaining to new hires for group. Managed personnel matters including reviews, problem resolution, and conflicts between personnel. Created training manuals and Standard Operating Procedures (SOP's) for both internal and external programs used.

LIMS Support: Provided support for the internal LIMS System of a commercial laboratory. Interacted with other laboratory locations (Pittsburgh and Houston) to provide advice and problem resolution. Assisted the Information Services Department maintain and repair problems within the database (Foxpro).

AECOM

Kelly A. Lurie, MPH

Education

MPH (Environmental Health), SUNY Albany, May 2015

MS, Geology (Geochemistry Focus), Rensselaer Polytechnic Institute, 2002

BS, Hydrogeology, Rensselaer Polytechnic Institute, 1999

AS, Environmental Studies, Hudson Valley Community College, 1996

Years of Experience

With AECOM: 22
With other firms: 0

Training and Certifications

HAZWOPER 40-Hour

HAZWOPER 8-Hour Supervisor

OSHA 30-Hour Construction

Practical Site Characterization and Remediation Techniques for DNAPLs and Their Associated Dissolved Phase

Ms. Lurie is responsible for environmental and geochemical analyses; data management and analyses; computer modeling; environmental sampling; document control, review, and QA/QC; development of Health and Safety Plans and Safe Work Plans; and project management and control during site remediation, including environmental construction and restoration. She has over twenty-two years of experience providing technical support, project support, technical review, and project management for the development of Remedial Investigations, Feasibility Studies, Proposed Remedial Action Plans, Records of Decision, Remedial Design studies, Site Management Plans, Quality Assurance Project Plans, Construction Completion Reports, and Final Engineering Reports per applicable regulations. She has taken the lead role in preparing project documents in compliance with the applicable requirements for clients including the New York State Department of Environmental Conservation (NYSDEC) and United States Army Corps of Engineers (USACE). She has also acted as a Safety, Health, and Environment (SH&E) Representative for her office, responsible for assisting AECOM SH&E regional management with matters at the local level.

Project Experience

Confidential Client, New York. Responsible for Construction Completion Report development and non-hazardous and hazardous waste management during implementation of remedial design that includes removal of contaminated soil from residential neighborhoods.

Lockheed Martin, Suffolk VA Glycol Leak, Suffolk, VA.
Project Manager for field exploratory activities to locate an underground leak from subsurface ethylene glycol lines by advancement of soil borings for the collection of soil and groundwater samples.

Lockheed Martin, Bloody Brook Site Management, Liverpool, NY.
Project Manager for site management activities following removal of soil and sediment from both residential and wetland areas contaminated with cadmium. Management activities include annual site inspections and adaptive management of developing constructed wetlands in consultation with the NYSDEC Division of Fish and Wildlife.

United States Army Corps of Engineers, Former Navy Depot, Scotia, NY. Responsible for providing technical and field support for the design and construction activities of a zero-valent iron permeable reactive barrier to promote the in-situ breakdown of chlorinated volatile organic compounds in groundwater. Alternate Site Supervisor/Site Safety Officer for these

activities. Co-authored the various project work plans, and lead authored the project Accident Prevention Plan in accordance with USACE Manual (EM) 385-1-1, the Quality Assurance Project Plan in accordance with the Uniform Federal Policy (UFP) for Implementing Environmental Quality Systems, and a Site Management Plan in accordance with NYSDEC's Technical Guidance for Site Investigation and Remediation (DER-10). Responsible for coordinating with the analytical laboratory and data validators and for completing overall project quality reviews.

United States Army Corps of Engineers, Former Camp Hero Remedial Investigation, Montauk, NY. Site Safety Officer for field activities during remedial investigation activities including the collection of soil borings, groundwater sampling, and wipe samples. Responsible for implementing the Community Air Monitoring Program (CAMP) developed for the field activities that included continuous monitoring for volatile organic compounds and particulates.

Lockheed Martin, West Branch of Bloody Brook Remedial Design and Construction, Liverpool, NY. Project.

Responsible for providing technical and field support as well as task management for a remedial design and construction program for removal of soil and sediment from both residential and wetland areas contaminated with cadmium. Task lead on development of the Site Management Plan and Final Engineering Report per DER-10.

Lockheed Martin, 241 Farrell Road Conceptual Pilot Study, Syracuse, NY. Field lead for subsurface investigation using a combination membrane interphase probe (MIP) and hydraulic profiling tool (HPT) to define stratigraphic and hydrogeological conditions as well as to better delineate a previously identified chlorinated organic solvents plume. Responsible for overseeing the subcontractor running the equipment as well as for interpretation of the field logs generated.

New York State Department of Environmental Conservation, Beaver Smelting, Fallsburg, NY. Project manager for site management activities for a small capped landfill containing metals contamination from a former aluminum recycling facility. Activities include monitoring levels of groundwater contamination; performing semi-annual landfill inspections; and preparation of a Site Management Plan and Periodic Review Report.

New York State Department of Environmental Conservation, Schatz Federal Bearings, Poughkeepsie, NY. Project manager for site management activities for a small capped landfill containing metals and volatile organic compounds. Activities include monitoring levels of groundwater contamination; performing semi-annual landfill inspections; and preparation of a Site Management Plan and Periodic Review Report.

New York State Department of Environmental Conservation, NY Air Brake, Watertown, NY. Deputy project manager for post remediation performance monitoring program. Responsible for evaluating residual contaminant levels for PCBs and cadmium in a stream system in order to provide documentation of the effectiveness of completed remediation. This evaluation ultimately identified a significant area of remaining contamination overlooked during prior remedial activities and resulted in additional cleanup activities at the site.

Constitution Pipeline, New York. Project manager for support of public outreach activities related to the Constitution Pipeline Project including development and maintenance of a database for tracking stakeholders'

information and records pertaining to outreach activities.

U.S. Army Corps of Engineers, Forest Glen Annex Remedial Investigation, Silver Spring, MD. Responsible for providing technical support for the preparation of an ecological risk assessment for potentially impacted water, soil, and sediment as part of a remedial investigation being completed under the Army's Installation Restoration Program and CERCLA.

Confidential Client, New York. Responsible for technical support and management and review of analytical data for an interim remedial measure design and implementation that includes removal of mercury contaminated soil from up to 150 residential properties.

New York State Department of Environmental Conservation, Midtown Shopping Center RI/FS, South Glens Falls, New York. Deputy Project Manager for the preparation of a remedial investigation and feasibility study to define the extent of previously identified soil and groundwater impacts resulting from dry cleaning activities.

General Electric, Hudson River Sediment Remediation 2010 Dredging, Contract 5 – Habitat Construction Proposal, Fort Edward, New York. Provided project control/support that included the coordination of deliverables to the client and document control.

Exide Corporation, Hi-Volume Ambient Air Monitoring, Muncie, Indiana. Responsible for sampling high volume ambient air monitoring systems for total lead. Also responsible for completing performance audits on the systems for quality control.

New York State Department of Environmental Conservation, Onondaga Lake RI/FS, Syracuse, New York. Environmental scientist for the preparation of a remedial investigation and feasibility study (RI/FS) at Onondaga Lake, a 3,000-acre polluted water body on the National Priorities List (NPL). Provided data management and analysis, field sampling, hydrogeologic investigations, risk assessments, and an analysis of interim remedial measures. Co-authored the re-write of the RI/FS remedial investigation report describing the nature and extent of contamination resulting from numerous inorganic and organic compounds, including mercury, BTEX, chlorinated benzenes, PAHs, PCBs, and dioxins/furans. Participated in surface water sampling throughout Onondaga Lake for a low-level mercury analysis to assess external sources of mercury and sediment resuspension.

Provided technical review and comments to New York State Department of Environmental Conservation (NYSDEC) on sediment capping and natural attenuation models included in the draft FS report prepared by the potential responsible party (PRP). The sediment capping model predicted breakthrough times and concentrations for key parameters, including mercury, chlorobenzene, and BTEX. Responsible for participating in numerous meetings with NYSDEC and the PRP to resolve technical issues within the FS report. Also assisted NYSDEC with the preparation of the proposed plan for remediation (PP) and with the package for the National Remedy Review Board (NRRB).

Attended public meetings, public availability sessions, and the presentation to the NRRB for the PP as technical support for NYSDEC. Assisted NYSDEC in the preparation of the record of decision (ROD) and the responsiveness summary (RS).

Responsible for providing technical review and comments to NYSDEC on work plans and data reports prepared by the PRP for the purpose of detailing

the environmental sampling and analyses completed during the predesign investigation (PDI). Task leader responsible for coordinating the update of the Onondaga Lake database.

New York State Department of Environmental Conservation, Geddes Brook / Ninemile Creek RI/FS, Syracuse, New York. Environmental scientist for re-write of remedial investigation and risk assessment reports for Geddes Brook / Ninemile Creek, a sub-site to the Onondaga Lake NPL site. Responsible for providing review and technical comments to NYSDEC based on RI and RA reports prepared by the PRP. Deputy task manager for re-writes of these documents based on NYSDEC disapproval. Co-authored the re-write of the RI/FS remedial investigation reports.

Responsible for data management and analysis and report preparation, including statistical summaries, sediment and soil screening, data summaries, and contaminant depth profiles. Authored sections and prepared graphics for documenting contaminant distribution in floodplain soils, sediment, and water and reviewed contaminant distribution maps for soil/sediment prepared in GIS. Provided oversight during a floodplain soil sampling event for the purpose of assessing the extent of contamination of Ninemile Creek floodplain.

Provided assistance for review of interim remedial measure document for Geddes Brook sediment and floodplain soils. Provided technical review and comments to NYSDEC on the draft FS report prepared by the PRP. Participated in meetings with NYSDEC and the PRP to resolve technical issues within the FS report. Provided technical comments on the supplemental FS prepared by the PRP. Co-authored the PP.

New York State Department of Environmental Conservation, Harbor Brook / Wastebed B RI/FS, Syracuse, New York. Provided NYSDEC with analysis of data collected during a preliminary site assessment (PSA) of the Harbor Brook / Wastebed B site, a sub-site of Onondaga Lake NPL site, to determine sampling requirements for remedial investigation. Also responsible for review and technical comments for RI work plan and draft RI report prepared by the PRP.

New York State Department of Environmental Conservation, Willis Avenue Chlorobenzene Site RI/FS, Syracuse, New York. Assisted in review of hydrogeological investigation and pumping test report for a sub-site of the Onondaga Lake NPL site. Evaluated hydraulic conductivities of the hydrogeologic units along the Onondaga lakeshore area, which is contaminated with BTEX and chlorinated benzenes. Analysis was based on pumping test data from the site using groundwater-modeling software, AQTESOLV. Provided review, technical comments, and verification of calculations for the remedial investigation and human health risk assessment reports prepared by the PRP.

New York State Department of Environmental Conservation, General Motors Former Inland Fisher Guide Facility and Ley Creek Deferred Media RI/FS, East Syracuse, New York. Provided technical comments to NYSDEC for supplemental remedial investigation and human health risk assessment reports prepared by the PRP.

New York State Department of Environmental Conservation, Wastebeds 1 through 8 RI/FS, Syracuse, New York. Provided technical comments to NYSDEC for the RI/FS Work Plan prepared by the PRP.

Multiple Clients, Multiple Projects, Indiana and Illinois. Responsible for collecting groundwater samples using various low-flow techniques as well as traditional bailing. Samples were collected for a wide suite of analytes

including VOCs, metals, and biological parameters.

Raytheon Technical Services Co., Former Naval Air Warfare Center, Indianapolis, Indiana. Responsible for the collection of paint chip samples and wipe samples for PCB analysis.

US Army Corps of Engineers - Kansas City District and USEPA - Region II, Reynolds Metals Site PRP Oversight Support, Massena, New York. Provided technical support for preliminary review of an in-situ sediment cap design.

US Army Corps of Engineers, Hudson River PCBs Superfund Site RRI/FS, New York. Responsible for preliminary analysis of metals and dioxins/furans sediment data to be used in assessing the development of resuspension and residual engineering standards for these compounds as a result of remedial dredging.

USEPA - Region II, Hiteman Leather Site, West Winfield, New York. Responsible for drilling oversight, soil classification, and soil sample collection.

Rensselaer Polytechnic Institute, New York. As graduate research assistant, participated in extensive ongoing research on the Hudson River, New York, and the NY/NJ Harbor, including field sampling and data management and analysis. Research involved characterizing pesticide contamination in the NY/NJ Harbor based on extensive sediment and fish data. Participated in the collection and processing of sediment cores in preparation for radionuclide dating. Responsible for maintaining the radionuclide gamma counter in the spectroscopy lab.

Publications and Presentations

Conference Presentations

Montione, R., Kelly Robinson, and Michael L. Spera. “Identification of Recent Markers for Sediment Dating in Onondaga Lake," poster presented by R. Montione at the Seventh Annual Onondaga Lake Scientific Forum, Liverpool, New York, November 11, 2005.

Robinson, K, R. Bopp, and D. Chaky. “Chlordane and DDT in the Hudson: Recent Trends,” poster presented by K. Lurie at the National Institute of Environmental Health Sciences conference: Persistent Chemicals: New Priorities, New Concerns, Bear Mountain, New York, September 29-30, 2004.

Publications

Montione, R., M. Spera, H. Chernoff, and K. Robinson. “Development of Sediment Remediation Goals for the Onondaga Lake, NY Superfund Site,” Remediation of Contaminated Sediments - 2007: Proceedings of the Fourth International Conference on Remediation of Contaminated Sediments, January 2007.

Kelly Robinson, "DDT and Chlordane in the Hudson River Basin," Masters thesis, Rensselaer Polytechnic Institute, Troy, New York, May 2002.

Kelly Lurie, Shedrack R. Nayebare, Zafar Fatmi, David O. Carpenter, Azhar Siddique, Daniel Malashock, Kamran Khan, Jahan Zeb, Mirza M. Hussain, Fida Khatib, and Haider A. Khwaja. “$PM_{2.5}$ in a Megacity of Asia (Karachi): Source Apportionment and Health Effects”. Atmospheric Environment, 202, pages 223-233, April 2019.

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Tim Verslycke, Ph.D.

Principal

Dr. Verslycke has 20 years of combined consulting and academic research experience in ecotoxicology and ecological risk assessment. His areas of expertise include ecotoxicology and ecological risk assessment; natural resource damage assessment; industrial and consumer product environmental safety assessment; and emerging contaminants. He has authored over 40 peer-reviewed journal articles and book chapters in these topic areas. Dr. Verslycke previously held an academic position in the Biology Department at the Woods Hole Oceanographic Institution (Woods Hole, MA). He previously served on the Steering Committee of SETAC's Global Endocrine Disrupter Testing and Risk Assessment Advisory Group and SETAC's Global Pharmaceutical Advisory Group. He also served on US EPA's Board of Scientific Counselors Safe and Sustainable Water Resources Subcommittee. He is a former president of the SETAC North Atlantic Chapter. He is also a former scientific advisor to the Center for Health and Environment of the Flanders Regional Government (Belgium) and a former member of the scientific committee of the Flanders Marine Institute (VLIZ, Belgium). He is a founding member and currently serves as president of the International Board of Environmental Risk Assessors (IBERA). IBERA established the first international certification program in ecological risk assessment.

Areas of Expertise

- Ecotoxicology & Ecological Risk Assessment
- Natural Resource Damage Assessment
- Product Stewardship & Sustainability
- Emerging Contaminants

Services

- Ecological Risk & NRD
- Water Resources
- Sustainability Consulting
- Renewables
- Climate Science
- Environmental Risk Assessment

Education

- Ph.D., Bio-Engineering/Applied Biological Sciences, Ghent University, Belgium
- M.S., Bio-Engineering/Environmental Technology, Ghent University, Belgium
- B.A., Bio-Engineering/Environmental Technology, Ghent University, Belgium

Selected Projects

Site Ecological Risk Assessments: Conducted ecological risk assessments at numerous US and international contaminated sites.

Ecological Injury Assessment and Causation Analysis: Conducted ecological injury assessments and causation analyses for ecological receptors exposed to a variety of chemical and non-chemical stressors at different sites.

Environmental Stewardship: For global companies, evaluated the environmental safety of their products to support sustainability goals.

Pharmaceutical Environmental Risk Assessments: For pharmaceutical companies, evaluated the environmental risk of their new drugs to support market approval, compliant with either European (EMA) or US (FDA) guidelines. As part of several assessments, oversaw the collection of additional environmental fate and toxicity test data.

Emerging Contaminants: In the context of a wide variety of projects, provided consulting and expert support related to the environmental safety of various contaminants of emerging concern, such as endocrine disruptors, pharmaceuticals, 1,4-dioxane, PFAS, microplastics, *etc.*

Endocrine Disruptors: For companies, trade associations, and government (US EPA), provided technical expertise related to the topic of endocrine disruption.

Selected Publications

Mebane, CA; **Verslycke, T,** *et al.* 2019. "Scientific Integrity Issues in Environmental Toxicology and Chemistry: Improving Research Reproducibility, Credibility, and Transparency." *Integr. Environ. Assess. Manag.* 15(3): 320-344.

Wait, D; **Verslycke, T.** 2019. "Expert Insight: Uniform data quality ecotoxicity assessment." In *Natural Resource Damages: A Guide to Litigating and Resolving NRD Cases* (Eds: Israel, BD; Marston, B; Daniel, L), American Bar Association, Chicago, IL, p213-215.

Marty, MS; **Verslycke, T,** *et al.* 2017. "Population-relevant endpoints in the evaluation of endocrine-active substances (EAS) for ecotoxicological hazard and risk assessment." *Integr. Environ. Assess. Manag.* 13(2):317-330.

Verslycke, T, *et al.* 2016. "Human health risks of triclosan in land-applied biosolids." *Environ. Toxicol. Chem.* 35(9):2358-2367.

Verslycke, T, *et al.* D. 2014. "The Chemistry Scoring Index (CSI): A hazard-based scoring and ranking tool for chemicals and products used in the oil and gas industry." *Sustainability* 6:3993-4009.

Peter E. Hollatz
Project Manager

Professional History

07/2001 - Present, AECOM

Education

BS, Civil and Environmental Engineering, Brown University, 2001

Registrations

Professional Engineer, Indiana
Professional Engineer, Illinois

Years of Experience

With AECOM: 22
With Other Firms: 1

Mr. Hollatz has over 22 years of experience performing environmental evaluations, remediation system design/implementation, and project management. His career has focused on the commercial/industrial sectors with emphasis on characterization and remediation of industrial facilities including aerospace manufacturing facilities, automotive parts manufacturing facilities, petroleum releases, and chlorinated solvent projects. As an engineering technical lead and project manager, Mr. Hollatz has executed projects in numerous states in state lead programs and federal lead programs. Extensive experience with aspects throughout the remediation process including: remedial strategy development, conceptual design (including pilot testing), detailed process and instrumentation design, design specifications, contractor selection process, field implementation, startup and optimization, life cycle operation and maintenance.

Experience

Carrier Corporation, Project Manager, Regulatory Strategy, and Remediation Design/Implementation, Syracuse, New York. Project manager and support regulatory project strategy at the Carrier facility in which the regulatory framework included elements from New York Department of Conservation (NYSDEC), New York Department of Health (NYDOH), United States Environmental Protection Agency (USEPA) Resource Conservation and Recovery Act (RCRA), and US Army Corps of Engineers.

Confidential Aerospace Client, Project Manager, Regulatory Strategy, Remediation Design/Implementation, Rockford, Illinois. Support regulatory project strategy at two Client properties in which the regulatory framework included elements from (USEPA RCRA, Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), Illinois Environmental Protection Agency (IEPA) Tiered Approach to Corrective Action Objectives (TACO), and IEPA Site Remediation Program (SRP). Design and implement Soil Vapor Extraction (SVE) system and groundwater pump and treat (GWPT) system to remediate chlorinated solvent impacts. Design included a portion of the treated water to be reused in facility processes.

Confidential Aerospace Client, Project Manager, Regulatory Strategy, Remediation Design/Implementation, Redmond, Washington. Support regulatory project strategy under the Voluntary Cleanup Program (VCP). Design and implement Sub-Slab Depressurization System (SSDS) and air sparge (AS)/SVE system to remediate chlorinated solvent impacts.
Confidential Automotive Parts Client, Regulatory Strategy and Remediation Design/Implementation, Dayton, Ohio. Support regulatory project strategy under USEPA CERCLA. Design and implement AS/SVE system to remediate chlorinated solvent impacts.

Attachment 2

Daily Calibration Form

AECOM Laboratory Certification #12995
Daily Field Instrument Quality Assurance Form

Site Name: _____

Project/Client: _____ **Project Number:** _____

Location: _____

Field Analyst(s): _____

Date: _____ **Start Time:** _____ **Finish Time:** _____

Instrument Information

Multi Meters calibrated by a rental company can be used for only 1 week. Sampling events extending beyond 1 week require a new rental unit with new calibration report.

Meter Type	Rental Period	Model	Certification #	Meter Serial #	Probe Serial #
Multi Meter					
Turbidity Meter					Not Applicable

Pre-Field Calibration and Quality Control Requirements Checklist

Prior to using instrument(s) in the field, field analysts must ensure that certain quality control procedures and calibrations have been completed for the instruments listed above. Records of the following, completed either by AECOM or the vendor, must be retained:

- ♦Weekly DO Winkler Titration ☐
- ♦5-Point Conductivity Curve ☐
- ♦Annual Conductivity Cell Constant ☐
- ♦Quarterly NIST Traceable Thermometer Check ☐
- ♦Solution Records ☐

Field Calibration/Calibration Checks

DO (Method SM 4500-O G)

100% Saturation (Calibration)	
Barometric Pressure (mmHg)	
Temperature (°C)	
Initial Reading	
Recalibrated Reading	

0% Saturation (Check)	
Solution Manufacturer & Lot #	
Solution Temperature (°C)	
Reading	
*Requires Recalibration	Y / N
Recalibrated Reading	

Calibration methods may differ between DO meters. Refer to instrument manual for calibration instructions. At minimum, a 1-point 100% saturation calibration (calibration mode), and a 0% Saturation check (measurement mode) shall be conducted prior to sampling.

Specific Conductance (Method 2510 B-11)

Standard Solution	Solution Manufacturer & Lot #	Solution Expiration Date	Solution Temperature (°C)	Reading ($\mu S \cdot cm^{-1}$)	*Requires Recalibration	Recalibrated Reading ($\mu S \cdot cm^{-1}$)
					Y / N	

ORP

Standard Solution	Solution Manufacturer & Lot #	Solution Expiration Date	Solution Temperature (°C)	Reading (mV)	*Requires Recalibration	Recalibrated Reading (mV)
					Y / N	

Turbidity (Method 2130 B): Report to the significant figures found in the table below.

Standard Solution (NTU)	Solution Manufacturer & Lot #	Solution Expiration Date	Reading (NTU)	*Requires Recalibration	Recalibrated Reading
				Y / N	
				Y / N	
				Y / N	
				Y / N	

Turbidity Range (NTU)	Report to the Nearest (NTU)
0-1.0	0.05
1-10	0.1
10-40	1
40-100	5
100-400	10
400-1000	50
>1000	100

AECOM

AECOM Laboratory Certification #12995
Daily Field Instrument Quality Assurance Form

pH (Method SM 4500-H^+B)

pH Calibration

Buffer Solution	Solution Manufacturer & Lot #	Solution Expiration Date	Solution Temperature (°C)	pH Value Input	Meter Adjusted Reading ±0.05 units	*Requires Recalibration	Recalibrated Reading
						Y / N	
						Y / N	
						Y / N	

Initial pH Check

Buffer Solution	Solution Manufacturer & Lot #	Solution Expiration Date	Solution Temperature (°C)	Reading ±0.1 units	*Requires Recalibration	pH Value Input	Recalibrated Reading
					Y / N		

3-hour pH Calibration Check

Time	Buffer Solution	Solution Manufacturer & Lot #	Solution Expiration Date	Solution Temperature (°C)	Reading ±0.1 units	*Requires Recalibration	pH Value Input	Recalibrated Reading
						Y / N		
						Y / N		
						Y / N		

*Field check readings should be within the accuracy listed by the manufacturer's specifications or per New Jersey regulations (NJAC 7:18), **whichever is more stringent**. Otherwise, the instrument must be recalibrated for that parameter. Refer to instrument manual for accuracy. Values provided below for instruments typically used by AECOM:

YSI 6-Series:		Horriba U-50 Series:	
DO	± 2% of the reading or ± 0.2 mg/L, whichever is greater	DO	± 0.2 mg/L
pH **	± 0.2 units	pH **	± 0.1 units
	**NJAC 7:18 requires that calibration values be accurate to ±0.05, and accuracy within ±0.1 units for the initial calibration check and ±0.2 units for 3-hour checks		
Specific Conductivity	± 0.5% of reading + 0.001 mS/cm	Specific Conductivity	± 1%
ORP	± 20 mV	ORP	± 15 mV
Turbidity	± 2% of the reading or ± 0.3 NTU, whichever is greater	Turbidity	± 5% of the reading or ± 1 NTU, whichever is greater

LaMotte 2020e/i Turbidity Meter:

±0.05 NTU or ±2% of reading whichever is greater, below 100 NTU

±3% of reading, above 100 NTU

Duplicate Analysis

Time	Parameter	Sample 1	Sample 2	RPD, %

Precision Acceptance Criteria:

DO	10%
pH	±0.1 SU
Specific Conductivity	1%
Turbidity	5%
Temperature	0.5° C

Attachment 3

SGS North America's Relevant Standard Operating Procedures

Standard Operating Procedure for Extraction of Various Matrices

Issue date: 11/01/2023
Revision: 11

SGS North America Inc.
5500 Business Drive
Wilmington, North Carolina 28405

Approved by:

J. Harrison Land, Last revised by — Date: 11/1/2023

Wade Maresh, Technical Manager — Date: 11/1/23

Jeannie Milholland, Quality Assurance Director — Date: 11/1/2023

(Official copies of final documents will contain all three signatures.)

TABLE OF CONTENTS

Revision History

- The following revisions were made effective on November 1, 2023:
  - Updated 8290 to 8290A and updated 1613 to 1613B throughout SOP.
  - Section 14 – removed reference to cleanup pumps as we no longer use them.
  - Added Table 15.1 for Air spike profiles.
  - Added Table 15.2 for Solid and Aqueous spike profiles.
  - Added sections 16.18 through 16.24.
- The following revisions were made effective on August 29, 2023:
  - Deleted CARB-429 referenced from SOP.
  - Added Section 5.2.3 for M23 PAH
  - Added Section 5.2.4 for M23 PCB
  - Added Section 5.2.5 for M23 PCDD/PCDF
  - Added Section 9.1.2 for bench sheet reference.
  - Section 9.7.11 – updated setting from "70" to "80"
  - Added Section 9.9.4 for M23
  - Updated Section 9.9.5 for M23
  - Section 17 – removed obsolete references and added references for M23, Horizon LIMS, APLIMS, and SGS template DC_560

- The following revisions were made effective on November 21, 2022:
  - Section 1.3 – added Project Manager
  - Section 4.2 – added PCBs and Pesticides
  - Sections 5.2, 5.5, and 5.6 – updated temperatures
  - Section 7.10 – updated sodium sulfate baking criteria
  - Section 9.1.3 – deleted abbreviated solids procedure and reference MI_13
  - Section 9.1.6 – clarified the requirement for TSS on water samples.
  - Section 9.1.6.7 – added requirement of NOT filtering DRBC samples.
  - Section 9.1.7 – removed the requirement of adding tetradecane.
  - New Section 9.2 references DC_353
  - Section 9.3.5 and 9.9.8 – added minimum.
  - Section 9.10 – added reference to Fish prep SOP DC_379
  - Removed section on semipermeable Membrane.
  - Section 11 added %TSS calculation.
  - Section 16 – updated XAD definition
  - Section 17 – added references to DC_364, DC_379, and MI_13
- The following revisions were made effective on March 21, 2022:
  - Updated section 7 for COA procedure

- Please refer to previous version of this SOP for revisions made on or before February 11, 2022.

1.0 Scope and Application

1.1 This method is intended to apply to all extractions performed at SGS-ILM, regardless of matrix. It is consistent with the Final Rule on the Methods Innovation Rule 40 CFR Parts 63, 268, et al.; June 14th, 2005; pp. 34538-34592.

1.2 To describe the procedures followed for the extraction of various matrices using Soxhlet Dean-Stark, Continuous Liquid-Liquid, temperature-assisted liquid-liquid extraction (TALEX) and/or other processes as applicable. This approach applies to air samples (e.g. XAD-2 resin, PUF, filters, wipes), soil, water, tissues and special matrices

1.3 Complex sample matrices may require special extraction techniques. Please see Technical Director, Project Manager, and/or Senior Chemist in these cases.

1.4 This SOP is based on SW-846 Methods 3540C/3520C/3510C/3580A

1.5 See Document DC_139 Appendix D for a full list of definitions.

2.0 Summary of Method

2.1 The primary objective of sample extraction is to quantitatively remove the analytes of interest from the matrix into an appropriate solvent, which will then be reduced in volume so that it can be cleaned up prior to analysis. For solid samples, the percent solid is determined. The samples are conditioned appropriately, depending on matrix. The samples are fortified with extraction standards and then extracted by a number of means, depending on which is appropriate for the matrix in question. Following extraction, additional conditioning may be necessary, after which the sample extracts are concentrated and prepared for cleanup.

3.0 Safety Precautions

3.1 Follow all appropriate safety guidelines, as laid out in our safety plans.

3.2 Gloves must be worn when handling samples, standards, and reagents.

3.3 Safety glasses must be worn at all times when working in the laboratory.

3.4 A lab coat must be worn while dealing with samples, standards, and reagents.

3.5 In the interest of safety and pollution prevention, all spills must be cleaned up immediately.

4.0 Interferences and Preventive Measures

4.1 Solvents, reagents, incorrectly cleaned glassware, and other extraction techniques may yield interference components in the final extract. See SOP MI_2 for glassware cleaning.

4.2 Three types of interferences may cause problems with this method. The three kinds of interferences are specific, non-specific, and quantitative, and each is considered below.

4.2.1 *Specific Interferences*: Specific interferences are PCDD/F's, PCB's, and/or Pesticides that appear in a final extract but did not originate with that sample. Two primary sources of specific interferences are cross-contamination from other samples and, less commonly, chemical reactions that form PCDD/F's, PCB's, and/or Pesticides during sample preparation procedures (e.g., extraction).

4.2.2 *Non-specific interferences*: Non-specific interferences are compound present in the final extract that creates false positive signals for PCDD/Fs (e.g., polychlorinated diphenyl ethers). Ideally, sample preparation removes these interferences, but analysts should always be wary of them. An exhaustive list of non-specific interferences may be found in – *Environmental Carcinogens: Methods of Analysis and Exposure Measurement Volume 11: Polychlorinated Dioxins and Dibenzofurans* (IARC Scientific Publications), C. Rappe (Editor), H. R. Buser (Editor), B. Dodet (Editor), I. K. O'Neill (Editor).

4.2.3 *Quantitative Interferences*: Quantitative interferences cause alterations in the measured instrumental response for one or more target analyte instrument responses. These interferences result from the presence of compounds that in the final extract that either suppress ionization or upset the lock mass correction of the instrument. For more detail on these types of interferences, see "Hybrid HRGC/MS/MS Method for the Characterization of Tetrachlorinated Dibenzo-p-dioxins in Environmental Samples." Y. Tondeur, W.J. Niederhut, S.R. Missler, and J.E. Campana, *Mass Spectrom.* **14**, 449-456 (1987).

4.3 Care should be taken in cleaning any extraction vessel or equipment that the sample or sample extract may contact during the extraction process.

4.4 Interferences co-extracted from samples vary from matrix to matrix and from sample to sample. Additional cleanup may be required as needed or specified by project.

4.5 Contamination or carry-over may occur by extraction of high level samples sequentially with clean or low level samples. Treat all samples as though they have the potential to cause cross-contamination, even when we are certain they cannot. Proper record keeping allows for review of how and in what order samples were extracted if a high level sample is discovered during analysis. This makes it possible to determine which samples may have carry over and need re-extraction.

4.6 Some PCBs are airborne. Use of a separatory funnel may result in contamination of samples with airborne PCBs. Lab method blanks should be monitored closely when using separatory funnels for PCBs.

5.0 Sample Collection, Preservation and Storage

5.1 Waste is disposed of in appropriate waste containers in accordance with the local, state and federal regulations. No sample or reagent is ever disposed of down a drain or in the trash. See document MI_278 for waste disposal, pollution prevention and spill clean-up.

5.2 Sample holding times and other storage requirements by method for solid samples are as follows:

5.2.1 Methods 1668A/B/C & 1613B: holding time is up to 1 year, store in the dark at 0-6° C) until extracted then frozen (≤-10°C) at client request until disposal

5.2.2 Method 8290A: holding time is 30 days from collection to extraction, store in the dark at 0-6° C)

5.2.3 PAH by Method 23 (air sample XAD, rinses and filters): holding time for extraction is within 30 days from collection. Extracts must be analyzed within 45 days. Store samples at ≤6 °C in the dark. Store extracts and archives at <-10 °C for up to 45 days.

5.2.4 PCB by Method 23 (air sample XAD, rinses and filters): holding time for extraction is within 1 year from collection. Extracts must be analyzed within 1 year. Store samples at ≤6 °C in the dark. Store extracts and archives at <-10 °C for up to 1 year.

5.2.5 PCDD/PCDF by Method 23 (air sample XAD, rinses and filters): holding time for extraction is within 1 year from collection. Extracts must be analyzed within 1 year. Store samples at ≤6 °C in the dark. Store extracts and archives at <26 °C for up to 1 year.

5.2.6 Method 1699: holding time is 1 year, store at 0-6° C) until extracted then frozen (≤-10°C) at client request until disposal

5.2.7 Extracts should be analyzed within 40 days for Semivolatile organics and organochlorine pesticides. Extracts should be analyzed within 45 days for PCDDs and PCDFs.

5.3 Water samples are collected in 1 L amber glass (2 L size for DRBC) containers following conventional sampling practices.

5.4 Soil samples are collected in 1 4oz amber wide-mouth glass container.

5.5 Sample holding times and other storage requirements by method for aqueous samples are as follows:

5.5.1 Methods 1668A/B/C & 1613B: holding time is up to 1 year, store in the dark at 0-6°C

5.5.2 Method 8290A: holding time is 30 days from collection to extraction, store in the dark at 0-6°C

5.5.3 Method 1699: holding time is 7 days, store at 0-6°C

5.6 Sample holding times and other storage requirements by method for tissue samples are as follows:

5.6.1 Methods 1668A/B/C & 1613B: holding time is up to 1 year, store in freezer at ≤-10°C

5.6.2 Method 8290A: holding time is 30 days from collection to extraction, store in freezer at ≤-10°C

5.6.3 Method 1699: holding time is 1 year, store in freezer at ≤-10°C

6.0 Equipment and Supplies

6.1 Soxhlet setup

6.1.1 Soxhlet body equipped with drain tube

6.1.2 Dean Stark

6.1.3 Condenser

6.1.4 500mL round bottom flask

6.2 Suitable personal protection equipment (PPE)

6.3 Clean stainless steel forceps

6.4 Aluminum foil

6.5 Drying Oven

6.6 Top-Loader Balance
6.7 Aluminum weight boats
6.8 Glass thimble (glass fritted at bottom)
6.9 Heating mantle
6.10 Chiller
6.11 Teflon Boiling chips
6.12 Concentrator
6.13 Table shaker
6.14 Graduated cylinder (various volumes)
6.15 Filtration device
6.16 Appropriate micropipette
6.17 Butcher knife
6.18 Meat grinder (Hobart or equivalent)
6.19 Fume hood
6.20 Freezer
6.21 Glass Beakers (various sizes)
6.22 40 or 60 ml vials
6.23 Diatomaceous earth (Hydromatrix™)
6.24 Borosilicate pipets, disposable, serological (various volumes)
6.25 Amber glass bottles, 1 L or 2.5 L(Teflon-lined screw cap)
6.26 separatory funnels varying sizes
6.27 Glass chromatographic columns
6.28 N-Evaporator, rocket evaporator or equivalent
6.29 Conical autosampler vials, 2mL
6.30 Glass fiber filters
6.31 Glass wool plugs
6.32 Glass Funnels
6.33 Rotary Evaporator or Vacuum Centrifuge
6.34 15mL test tubes
6.35 Tissuemizer

6.36 Stainless Steel Spatula

6.37 pH Paper

6.38 CLLE extraction bodies

6.39 CLLE rack with condenser and water lines

6.40 Drying Adaptor

6.41 Water Heater

6.42 Snyder Column

6.43 Various sized micron filter papers (2 µm, 1 µm, 0.7 µm and 0.45 µm)

6.44 4 or 8 oz. jars

6.45 Semipermeable membrane

6.46 Membrane crimper

6.47 Heat sealer

6.48 Carbon Pellets

6.49 Carbopak carbon

6.50 Side arm Erlenmeyer flasks

6.51 Büchner funnels

6.52 Vacuum pump

6.53 Chlorine test strips

6.54 AX-21

6.55 Sonicator

6.56 Appropriately sized round bottom

6.57 Custom Talex column

6.58 Boiling stones

6.59 Glass beads

7.0 Standards and Reagents

7.1 Hard copies of COAs are saved in the Supply Prep Logbook (SPL) behind the appropriate SPL page. The COA is labeled with the appropriate SPL ID. Anyone using a new lot for the first time is responsible for retrieving the COA from the vendor web site if not supplied by the vendor and saving it in the SPL book. Solvent kegs will follow the same rules and are kept in the Solvent tracking Logbook (STL).

7.2 Note: Not all reagents are used in every extraction type.

7.3 Hexane Highest available purity.

7.4 Toluene Highest available purity.

7.5 Isooctane Highest available purity

7.6 Methylene chloride Highest available purity.

7.7 Sulfuric acid, concentrated

7.8 Tetradecane Highest available purity.

7.9 Methanol Highest available purity.

7.10 Anhydrous Sodium Sulfate, baked at 400°C for at least 4 hours, stored in container with lid, and labeled with tracking number and expiration date.

7.11 DI water

7.12 Cleaned DI water/Distilled water

7.13 Acetone Highest available purity.

7.14 Silica gel. Highest purity grade.

7.15 Florisil

7.16 Pre-purified nitrogen gas.

7.17 Sodium Hydroxide. Highest available purity.

7.18 Diethyl Ether

7.19 Analytical standards, in toluene, (Cambridge Isotope Laboratory, Woodburn, MA, Wellington Laboratories, or other qualified vendor). See Table 3 of the dioxin/furan analysis SOP DC_364 for details on composition of standard solutions.

7.20 Extraction Standards (20ul@100pg/uL in Toluene for D/F, 20ul@100pg/ul in nonane for PCB) See project spike profile for exact details.

7.21 Sodium Thiosulfate, 99.99+%, powder

8.0 Calibration

8.1 Pipette Calibration

8.1.1 Refer to SGS document MI_46 for pipette and syringe calibration procedure.

8.2 Balance Calibration Verification

8.2.1 Refer to SGS document MI_1 for balance verification procedure.

9.0 Sample Preparation

9.1 Sample Examination

9.1.1 When first opening a sample, examine the sample for any qualities that may affect reliable analysis. A strong chemical odor may indicate that a sample will contain particularly high concentrations of target analytes or laboratory contaminants. It may also indicate potential interference problems, such as high fuel content. Note any large pebbles, rocks, twigs, shells and other matter that interferes with representative subsampling. For aqueous samples, note color and solids content. Record any observations you make that you believe may affect the analysis.

9.1.2 All observations will be recorded on bench sheets (see template DC_560).

9.1.3 If you have reason to suspect that a sample may contain unusually high concentrations of target analytes, make note of this fact in the documentation accompanying the sample and consult the Technical Director, Project Manager, and/or Senior Chemist for instructions on how to proceed.

9.1.4 Determine the percent solids on all soil and sediment samples following SGS SOP MI_13. Percent solids determinations are not necessary for air matrices. For other matrices, check with project management for percent solids requirements if not included on the initiation form.

9.1.4.1 All samples are extracted on a wet weight basis. For solid samples where a percent solids determination has been made, adjust the sample amount for extraction to yield a dry weight equivalent (DWE) based on the percent solids, usually defaulting to 10g DWE .

9.1.4.2 Ensure all samples are homogenized.

9.1.4.3 The lab will use a one gram dry-weight equivalent sample size for sediment samples believed to contain unusually high levels of PCBs (>80 ppb). In addition, in these cases, the lab will spike 10 ng of the ES instead of the usual 2 ng. Finally, the lab will use a final volume of 100 µL of nonane instead of the usual 20 µL. These enhancements are designed to prevent unnecessary extraction of

samples and saturation of the HRMS detector).

9.1.5 Determination of percent oil or lipids if required.

9.1.5.1 Extract the entire contents of the sample using a soxhlet extraction apparatus.

9.1.5.2 Remove any residual water from the extract using sodium bisulfate to dry the sample.

9.1.5.3 Filter solid particulate from the extract and concentrate the sample down to 10mL.

9.1.5.4 Using a class A, 1.00mL syringe, transfer 1mL of the extract to a pre-tared aluminum weigh boat.

9.1.5.5 Concentrate the extract aliquot to “dryness” at room temperature.

9.1.5.6 Record the weight of the aluminum weigh boat and oil residue using an analytical balance accurate to 0.002g (+/- 0.0002g).

9.1.5.7 Place the weigh boat in a vacuum desiccator and allow to concentrate for at least 1 hour.

9.1.5.8 Re-weigh the sample and weigh boat to ensure that no additional evaporation has occurred, and the weight remains stable. If the second weight does not match within +/- 0.0002 of the first weight, repeat the process starting at 9.1.4.7 until criteria is met.

9.1.5.9 The residual oil weight is calculated by subtracting the weight of the boat from the total weight of the boat with dried oil residue.

9.1.5.10 Multiply the oil residue weight by 10 to correct the weight to the original sample volume, and calculate the oil or lipid content to the nearest three significant figures using calculation shown in section 11.2

9.1.6 Sub Sampling (if needed)

9.1.6.1 Water

9.1.6.1.1 Homogenize sample to incorporate and evenly distribute particulate matter.

9.1.6.1.2 Use a power shaker if desired (consider the possibility of incorporating airborne PCBs).

9.1.6.2 Soils and Sediments

9.1.6.2.1 The sample should be stirred thoroughly to ensure homogeneity. The sample is homogenized and a representative sample is taken after homogenization.

9.1.7 Aqueous or Effluent Samples (nominal volume of 1 liter)

9.1.7.1 If the sample contains large amounts of visible particulate matter determine the suspended solids (TSS) content.

9.1.7.2 DRBC program samples must not be filtered.

9.1.7.3 All paper mill effluent samples must be filtered and the isolated solids extracted by SPE and SDS.

9.1.7.4 To determine the percent TSS filter 10 mL of sample through 0.45µm glass fiber filter.

9.1.7.5 Place in oven at 110°C ±5°C for at least one hour.

9.1.7.6 If the solid load is <1.0 percent, the sample may be extracted using the TALEX or CLLE approaches.

9.1.7.7 The details for these procedures are given below. The decision to follow one particular approach over the others is dictated by the client's requirements, the type of samples undergoing analysis, and the expertise of the laboratory staff.

9.1.7.8 If the resulting suspended solids analysis is ≥1.0 percent solids, extract the filter as a solid, extract the water and combine the extracts. If the particulate appears to be easily removed by filtration, perform SPE-C as outlined in section 9.5.

9.1.8 Extraction Solvents and heating mantle temperature settings:

9.1.8.1 D/Fs solids: Toluene

9.1.8.2 PCBs/USVs/PAHs solids: Hexanes

9.1.8.3 Tissues (moderate to high % lipid content)- Hexanes. EU Tissues- Hexanes: Acetone.

9.1.8.4 The heater should be set to approximately~50% for methylene chloride and hexanes, approximately 65% for toluene.

9.2 See SGS document DC_353 for fortification procedure.

9.3 **Soils & Sediments by Soxhlet and Soxhlet/Dean-Stark (SDS)**

9.3.1 Weigh the samples, and batch QC (LMB/OPR), in a clean glass thimble or sample jar containing Hydromatrix™ or sodium sulfate. Batch QC is treated exactly as a sample, including exposure to all glassware, equipment, solvents, reagents, internal standards, and labeled compounds. Perform the following steps for each sample.

9.3.2 Stir the thimble's contents to thoroughly mix the sample with the Hydromatrix™ or sodium sulfate; dispersing the sample across the surface of the inert material. This step will have the secondary effect of absorbing the liquid present in the sample.

9.3.3 Transfer the sample to a extraction thimble and place in a Soxhlet Extraction apparatus. Add the Dean-Stark adapter if sodium sulfate is not used.

9.3.4 Fortify the samples with the appropriate extraction standards. Extract the samples by Soxhlet or SDS by adding 300-400 mL of the appropriate solvent to a 500 mL Round Bottom Flask containing Teflon ™ chips. Extraction must be for a length of time sufficient to effect full recovery of extraction standards. By default, the extraction time is 16 hours, although shorter times may be used if they have been previously demonstrated through validation to provide adequate performance.

9.3.5 During the extraction, verify that the solvent cycles normally. The solvent must cycle through the system a minimum of five times each hour.

9.3.6 Drain any water from the Dean-Stark as appropriate to prevent water from returning to the boiling flask.

9.3.7 At the end of the reflux cycle time, proceed with sample concentration as described below.

9.3.8 Drain the solvent present in the DS side arm directly to solvent waste. Open stopcock on thimble holder and continue to drain solvent to waste until approximately 25 mL of solvent is remaining in the round bottom flask.

9.3.9 Turn off the heater and allow the solvent to continue to concentrate as low as approximately 5 mL. Do NOT allow to go "dry" as this will decrease target analyte recovery.

9.3.10 Allow the flask to cool down.

9.3.11 Transfer the extract to an appropriate sample container, spike with the required cleanup standards, and proceed to concentration/solvent exchange as described in DC 365 Fractionation (Cleanup Procedures).

9.4 **Continuous Liquid-Liquid Extraction** (using extraction bodies with Snyder columns)

9.4.1 Remove samples from designated storage area and allow them to come to room temperature.

9.4.2 Make sure the individual water supply valves on the CLLE rack are closed.

9.4.3 Turn on the main pump valve to "on".

9.4.4 Add one large boiling chip to each concentrator.

9.4.5 Attach the extraction body, drying adapter (with sodium sulfate) and concentrator to the CLLE rack.

9.4.6 Attach the water supply and return lines to the concentrator, and open the valves.

9.4.7 Turn on the water heater and allow the temperature to come up to 160 ± 5°C.

9.4.8 Make sure the chiller is running at the proper temperature (10 ± 3°C).

9.4.9 Add approximately 250 mL of methylene chloride to each extraction body.

9.4.10 Test for residual chlorine and record the pH of the sample. If residual chlorine is present, then add 80 mg sodium thiosulfate per liter of water. If pH is > 9 for NPDES permit compliance

samples then adjust the pH to < 9 using sulfuric acid.

9.4.11 Place sample jar on a balance and tare the scale.

9.4.12 Add the contents of the sample bottle to the extraction body, use 1L of deionized water for batch QC samples (LMB/OPR). Batch QC is treated exactly as a sample, including exposure to all glassware, equipment, solvents, reagents, internal standards, and labeled compounds.

9.4.12.1 Use the entire sample container for all DRBC samples, use 2L of distilled water for DRBC Sample QC

9.4.13 Reweigh the sample jar on the tared scale and record the weight of the displaced sample volume.

9.4.14 Rinse the jar with Methylene Chloride and add to the extraction body.

9.4.15 Spike the sample with extraction standards as listed on the spike profile.

9.4.16 Attach the condenser to the extraction body and open the stopcock.

9.4.17 Allow the samples to extract for 16 to 18 hours, as validated. South Carolina samples will be extracted for 24 hours.

9.4.18 When the extraction is complete, close the stopcock and allow the extract to concentrate down to approximately 10 mL.

9.4.19 Turn off the water heater.

9.4.20 Turn off the power to the water pump.

9.4.21 Crack open the individual water return valves to allow air in, and water to drain out.

9.4.22 Close the individual water supply and return valves.

9.4.23 Transfer the extract to an appropriate sample container, spike with the required cleanup standards, and proceed to concentration/solvent exchange as described in DC 365 Fractionation (Cleanup Procedures).

9.4.24 Properly dispose of the remaining sample water and Methylene Chloride.

9.5 Separatory Funnel Extraction

9.5.1 Prepare drying funnels by packing the neck of a glass funnel with

glass wool.

9.5.2 Add approximately 10-15 g of Na_2SO_4 on top of the glass wool and position over an appropriately labeled collection vial (containing keeper solvent if required).

9.5.3 Test for residual chlorine and record the pH of the sample. If residual chlorine is present, then add 80 mg sodium thiosulfate per liter of water. If pH is > 9 for NPDES permit compliance samples then adjust the pH to < 9 using sulfuric acid.

9.5.4 Place the sample bottle on a balance and tare the scale.

9.5.5 Add the contents of the sample bottle to the separatory funnel, reweigh the bottle and record weight of the displaced sample volume.

9.5.6 Rinse the jar with Methylene Chloride and add to the extraction body.

9.5.7 Use 1L of deionized water for batch QC samples (LMB/OPR). Batch QC is treated exactly as a sample, including exposure to all glassware, equipment, solvents, reagents, internal standards, and labeled compounds.

9.5.8 Record the pH of the sample on the extraction sheet.

9.5.9 Add extraction standard to the sample, shake vigorously and allow it to integrate.

9.5.10 Add 60 mL of methylene chloride to the separatory funnel.

9.5.11 Shake for at least 2 min, periodically purging the separatory funnel of any excess pressure.

9.5.12 Allow the sample-solvent solution to settle out and form a distinct interface.

9.5.13 Drain the solvent through the sodium sulfate funnel into the collection vial.

9.5.14 Repeat the extraction twice more.

9.5.15 Spike with the required cleanup standards and proceed to concentration/solvent exchange as described in DC 365 Fractionation (Cleanup Procedures).

9.6 **SPE-C**

9.6.1 Obtain the weight of the sample and container. Transfer the

sample to the extraction flask and re-weigh the empty container. Determine and record the weight of the sample by difference.

9.6.2 For composites of pulp and paper acid/alkaline aqueous samples, composite according to the ratio or amount specified in the COC.

9.6.3 Measure pH and residual chlorine using test strips. Record the information. If residual chlorine is present, then add 80 mg sodium thiosulfate per liter of water.

9.6.4 Fortify the sample with the ES (along with Ax for the OPR or MS/MSD when requested).

9.6.5 If the samples are for 2378-TCDD only add 1 g of the carbon pellets (approximately 10 pellets). Pellets can be used for large volume samples also (i.e.. 4L)

9.6.6 If the samples are for a full list analysis add 1gof the carbo-pak carbon.

9.6.7 Allow the sample to agitate on the platform shaker for 2 hours(4 hours for large volume samples).

9.6.8 Filter through a 2 µm, 1µm, 0.7 µm filter then a 0.45 µm filter using an Erlenmeyer flask equipped with a Buchner funnel. Recover the filters and any cake/carbon pellets.

9.6.9 Rinse the bottle with toluene (10-25 mL) and add this rinse to the SDS toluene for the extraction of the solid catches.

9.6.10 Perform an SDS extraction on the filters/solids using toluene for 16 hours. Watch cycling for at least 1 hour to insure proper cycling, no loss of solvents, leakage, broken glassware.

9.6.11 Transfer the extract to an appropriate sample container, spike with the required cleanup standards, and proceed to concentration/solvent exchange as described in DC 365 Fractionation (Cleanup Procedures).

9.7 Aqueous Samples other than P&P Filtrates

9.7.1 The following procedure is not applicable for South Carolina compliance samples.

9.7.2 Use the custom-designed TALEX column.

9.7.3 Prime the column with a few mL (usually 2-3 mL) of DI water.

9.7.4 Add 3 mL of toluene for PCDD/Fs or 3 mL isooctane for PCBs.

9.7.5 Add the demister in the mid-section of the column (glass beads) and top the column with aluminum foil.

9.7.6 Obtain the weight of the sample and container. transfer the water sample to the round bottom flask and re-weigh the empty container. Determine and record the weight of the sample by difference.

9.7.7 Measure pH and residual chlorine using test strips and record in the paperwork. If residual chlorine is present, then add 80 mg sodium thiosulfate per liter of water.

9.7.8 Fortify the aqueous samples with the extraction/analytes standards directly into the round bottom.

9.7.9 Add several boiling stones (Do not use Carborandum). Make sure there are enough stones sitting at the bottom of the flask. Floating chips do not regulate boiling.

9.7.10 Allow chiller water to circulate through the condenser making sure to check for leaks and /or cracks.

9.7.11 Start heating by turning the knob to position “80” on the thermo controllers.

9.7.12 Record the time from the moment the water starts boiling. Allow the boiling to go on for 2-4 hours.

9.7.13 After 2-4 hours, turn off the heat and allow the system to cool down for approximately 30 minutes.

9.7.14 Open the stopcock on the condenser and slowly drain the water layer to waste.

9.7.15 Drain the toluene or isooctane into a 60-mL vial.

9.7.16 If necessary, tip the column to draw out most of the organic solvent.

9.7.17 Rinse the inside of the column with 2x3 mL of toluene for PCDD/Fs (or hexane for PCBs); draining into the vial with the original extract.

9.7.18 Spike with the required cleanup standards and proceed to concentration/solvent exchange as described in DC 365 Fractionation (Cleanup Procedures).

Note: If the sample is a clean water sample (e.g., drinking water) additional cleanup is not necessary.

9.8 Oil Samples

9.8.1 Acid Partition

9.8.1.1 Weigh 1 g of oil (fish oil) into a VOA vial for PCBs or 5 g of fish oil if the analysis is for dioxins.

9.8.1.2 Dilute the oil with approximately 30 – 40 mL of hexanes.

9.8.1.3 Spike the diluted oil with extraction standards.

9.8.1.4 Perform an acid partition with concentrated H_2SO_4 until the solution appears clear.

9.8.1.5 Transfer the hexanes layer into a clean vial.

9.8.1.6 Wash the hexanes layer with distilled water.

9.8.1.7 Run the hexanes layer through Na_2SO_4 to remove any residual water.

9.8.1.8 Spike with the required cleanup standards and proceed to concentration/solvent exchange as described in DC 365 Fractionation (Cleanup Procedures).

9.8.2 Carbon Column

9.8.2.1 Weigh out the required amount of sample into a 60 mL vial. Use an equal amount of hexane for the LMB and OPR.

9.8.2.2 Spike the samples with the extraction standards as listed on the spike profile.

9.8.2.3 If the sample is significantly more viscous than hexane, dilute the sample with hexane (usually 1:1).

9.8.2.4 Pack a glass column with glass wool, 3-4 cm sodium sulfate, 3-4 cm furnaced silica, approximately 0.5 grams of 5% AX-21, 3-4 cm furnace silica, 3-4 cm sodium sulfate and a glass wool plug.

9.8.2.5 Secure the column in a clamp with the "clean" end down and place a "waste" beaker underneath it.

9.8.2.6 Pre-elute the column with 20mL toluene, followed by 10mL hexane and allow to drip

into the waste beaker. After the solvent has eluted, flip the column so that the "clean" side faces up.

9.8.2.7 Transfer the sample onto the column.

9.8.2.8 When the entire sample has been transferred, rinse the sample beaker two times with 1 mL hexane and transfer to the column.

9.8.2.9 Continue to rinse the column with hexane until all the oil has been rinsed off the column (approximately 10 mL).

9.8.2.10 Remove the waste beaker and flip the column.

9.8.2.11 Place a 60mL vial under the column and elute with 50 mL toluene.

9.8.2.12 Concentrate the toluene,solvent exchange to hexane if necessary, and spike with cleanup standards.

Note: This extraction procedure was designed to accommodate large amounts of oil. For smaller amounts of oil, the sample may be added directly to a cleanup column.

9.8.3 Florisil Column

9.8.3.1 Weigh out the required amount of sample (nominally 20g for Dioxin analysis) into a 250 mL flask and add 50 mL of Hexane. Use an equal amount of hexane for the LMB and OPR.

9.8.3.2 Spike the samples with the extraction standards as listed on the spike profile.

9.8.3.3 The use of mild heat and sonication may be required to speed the dissolving of the oil in the solvent.

9.8.3.4 Pack a glass column with glass wool, 2 g salt, 3 g of florisil, and 1 g of salt.

9.8.3.5 Secure the column in a clamp.

9.8.3.6 Position a waste collection beaker.

9.8.3.7 Pre-elute the column with 30 mL hexane and allow drip into the waste beaker.

9.8.3.8 Transfer the sample onto the column.

9.8.3.9 When the entire sample has been transferred, rinse the sample beaker with 2 mL hexane and transfer to the column.

9.8.3.10 Continue to rinse the column with hexane until all the oil has been rinsed off the column (approximately 60 mL).

9.8.3.11 Remove the waste beaker and position a sample collection flask.

9.8.3.12 Elute the sample with 240 mL methylene chloride.

9.8.3.13 Concentrate the methylene chloride, solvent exchange to hexane and spike with cleanup standards.

Note: This extraction procedure was designed to accommodate large amounts of oil (5-20 g). For smaller amounts of oil, the sample may be added directly to a cleanup column.

9.9 Air Samples

9.9.1 Collect all the sample components.

Note: Usually, samples being prepared by EPA Method 23 are comprised of a filter, an XAD-2 resin trap, an acetone rinse for both the front and back halves, a toluene rinse for both the front and back halves, and sometimes a methylene chloride rinse for both the front and back halves. Method 0023A samples contain the same components, but the method requires the filter and front half rinses to be extracted and analyzed as a separate sample from the XAD-2 resin trap and back half rinses. For ambient air samples (Method TO-9A), a PUF and filter combination is typical.

For a Method 23 analysis (only), photograph the filter and XAD-2 resin trap together for documentation purposes prior to combination. Take a photograph of each sample's components immediately before unpacking and combining in the thimble. Follow the steps below as written. If the analysis is by Method 0023A, follow the steps below except for combining the resin and filter – these components (with their respective rinses) constitute two separate samples. Combine resin and filter in glass SDS thimble.

9.9.2 See Table 15.1 for air spike profiles

9.9.3 If the client used spiked Carbon Rinse Jars, the acetone rinse jars will have TS spike in them.

9.9.3.1 To extract rinse jars, add methanol and extraction solvent (toluene or hexane). Then take pieces of glass wool and clean the inside of the jar then place glass wool into extraction thimble. Repeat this until the jar is completely clean. Finish by rinsing the jars with the extraction solvent (hexane or toluene) and pour it into the extraction thimble.

9.9.4 Concentrate the rinses to less than 5 mL using a rotovap or by other appropriate technique.

9.9.5 Fortify the filter with the appropriate filter spikes (FS for D/F and AS for PCB and PAH), and place into the extraction thimble.

9.9.6 Add the XAD-2 resin to the extraction thimble, followed by the concentrated solvent rinses.

9.9.7 Use the glass wool that was packed into the XAD-2 trap to collect any stray XAD-2 particles and solvent and place the glass wool plug in the thimble on top of the resin.

9.9.8 Into the thimble, spike the appropriate amount of Extraction Standards, as indicated in the project documentation.

9.9.9 Assemble the DS and condenser components. Extract the samples using SDS by adding 300-400 mL of an appropriate solvent to a 500 mL Round Bottom Flask containing Teflon™ boiling chips. Extraction must be for a length of time sufficient to effect full recovery of extraction standards. By default, the extraction time is 16 hours, although shorter times may be used if they have been previously demonstrated through validation to provide adequate performance.

9.9.10 During the extraction, verify that the solvent cycles normally. The solvent must cycle through the system a minimum of five times each hour.

9.9.11 Drain any water from the Dean-Stark as appropriate to prevent water from returning to the boiling flask.

9.9.12 At the end of the reflux cycle time, proceed with sample

concentration as described below.

9.9.12.1 Drain the solvent present in the DS side arm directly to solvent waste. Open stopcock on thimble holder and continue to drain solvent to waste until approximately 25 mL of solvent is remaining in the round bottom flask.

9.9.12.2 Turn off the heater and allow the solvent to continue to concentrate to a minimum of approximately 5 mL. Do NOT allow to go "dry", as this will affect target analyte recovery.

9.9.12.3 Allow the flask to cool down.

9.9.12.4 Transfer the extract to an appropriate sample container, spike with the required cleanup standards, and proceed to concentration/solvent exchange as described in DC 365 Fractionation (Cleanup Procedures).

9.9.13 Split the extract per the project instructions, archive as necessary.

9.9.14 Store the archived extract at room temperature in the vial with a Teflon™-lined screw top.

9.10 **Tissue Samples**: Tissue samples received as whole material are processed in accordance to DC_379 (Preparation of Fish Tissue samples for HRMS/ID analyses).

9.10.1 Fortify the sample with ES directly.

9.10.2 Mix the sample with 20-25 g of sodium sulfate, stir until a dry, free-flowing consistency is achieved, adding more Hydromatrix™ or sodium sulfate as needed. Be careful not to exceed an amount that can fit into the thimble for the sample size used.

9.10.3 Extract by Soxhlet, or soxhlet Dean-Stark using hexane for 16h (or other duration previously demonstrated when appropriate).

9.10.4 For samples exceeding 50g, implement the following additional steps:

9.10.4.1 After spiking the sample and thoroughly integrating the spike, split the sample across enough thimbles such that no thimble holds

more than 50g total.

9.10.5 At the end of the reflux cycle time, proceed with sample concentration as described below.

9.10.5.1 Drain the solvent present in the DS side arm directly to solvent waste. Open stopcock on thimble holder and continue to drain solvent to waste until approximately 25 mL of solvent is remaining in the round bottom flask.

9.10.5.2 Turn off the heater and allow the solvent to continue to concentrate to a minimum of 5 mL. Do NOT allow to go "dry", as this will affect target analyte recovery.

9.10.5.3 Allow the flask to cool down. If multiple Soxhlet bodies were used, combine the extracts prior to concentration.

9.10.5.4 In case the tissue contains sediment particles, it is recommended to follow the hexane (-acetone) extraction with a toluene-acetone extraction.

9.10.5.5 Transfer the extract(s) to an appropriate sample container, spike with the required cleanup standards, and proceed to concentration/solvent exchange as described in DC 365 Fractionation (Cleanup Procedures).

9.11 **SPME Fibers**

9.11.1 Alternative spiking schema (e.g., use of FS as an ES) may be instituted to facilitate best scientific practice on customer request.

9.11.2 SPME Fibers will be extracted by sonication, as follows:

9.11.3 **Water fraction**: Pour off water into a 60mL vial. Extract 3 times serially with 5 mL Hexane. Combine with the hexane extract of the fibers. Alternatively, if the liquid portion is too small to be transferred to a separate vial for extraction, the aqueous portion may be co-extracted by sonication with the fiber portion.

9.11.4 **Fibers:** Leave fibers and aqueous portion, if necessary, in the

vial which it was received. Spike with ES solutions, per project specifications. Fill the vial (at least to fully cover the fibers) with hexane and sonicate for at least 30 minutes. As soon as possible after sonication, transfer the hexane extract to a new 60 mL vial and rinse the vial again with additional hexane after transfer to ensure complete recovery of analytes and standards.

9.11.5 If necessary, and not already done, combine the extracts and remove any residual water by separation or addition of sodium sulfate.

Transfer the extract(s) to an appropriate sample container, spike with the required cleanup standards, and proceed to concentration/solvent exchange as described in DC 365 Fractionation (Cleanup Procedures). Depending on the fiber composition and extract cleanliness, cleanup of the extract may be unnecessary and is optional.

9.12 CONCENTRATION, CLEANUPS and TRANSFERS

9.12.1 See SOP DC_365

10.0 Analytical Procedure

10.1 See the SOP for the determinative method(s) for analytical procedures.

11.0 Details of Calculations

11.1 Total Suspended Solids (TSS):

$$\%TSS = \frac{A-B}{V} \times 100$$

where,

A = weight after drying in grams (filter + dried residue)
B = weight of filter in grams
V = Total volume filtered in mL

11.2 Oil/Lipid Content:

$$\%Oil/Lipid = \left(\frac{final - tare}{initial - tare}\right) \times 100$$

where,

final = weight after drying
initial = initial weight of sample
tare = weight of pan

12.0 Quality Control Requirements

12.1 Data outside of QC limits may be addressed by one or more of the following options:

12.1.1 Re-preparation and re-analysis of sample

12.1.2 Client notification

12.1.3 Discussion and qualification of data by case narrative

12.1.4 Re-sampling and reanalysis (client decision)

12.2 Data outside of QC limits may be reported if directed by the client. It must be qualified by a case narrative detailing the QC problems with advice on the usability of the data.

12.3 See the SOP of the associated determinative method(s) for specific requirements.

13.0 Data Review and Reporting Requirements

13.1 Refer to SGS document MI_141, Data review SOP

14.0 Preventative Maintenance

14.1 Pipettes are calibrated quarterly.

14.2 Balance verification performed daily or when in use

15.0 Tables

15.1 Air Spike Profiles

XAD Pre-Spikes	Pipette volume	Standard Concentration	Total concentration	Split Factor
PCDD/F SS	40 uL	40-100 pg/uL	1600-4000 pg	½
PCB SS	40 uL	100 pg/uL	4000 pg	½
PAH SS	80 uL	500 pg/uL	40,000 pg	¼
Pre-Extraction Filter Spikes				
PCDD/F FS	40 uL	40-100 pg/uL	1600-4000 pg	½
PCB AS	40 uL	100 pg/uL	4000 pg	½
PAH AS	80 uL	500 pg/uL	40,000 pg	¼
Extraction Standard				
PCDD/F ES	40 uL	100 pg/uL	4000 pg	½
PCB ES	40 uL	100 pg/uL	4000 pg	½
PAH ES	80 uL	500 pg/uL	40,000 pg	¼
Alternate Cleanup Standard				
PCDD/F AS	40 uL	100 pg/uL	4000 pg	½
Injection Standard				
PCDD/F JS	20 uL	100 pg/uL	2000 pg	½
PCB JS	20 uL	100 pg/uL	2000 pg	½
PAH JS	80 uL	125 pg/uL	10,000 pg	¼

Spiking profile for standard air project; modifications will be notated in specific sample instructions if profile changes.

15.2 Solid and Aqueous Spike Profiles

Solid and Aqueous Spike Profile	Pipette volume	Standard Concentration	Total concentration	Split Factor
Extraction Standard				
PCDD/F ES	20 uL	100 pg/uL	2000 pg	No
PCB ES	20 uL	100 pg/uL	2000 pg	No
PAH ES	20 uL	500 pg/uL	10,000 pg	No
Cleanup Standard				
PCDD/F CS/SS	20 uL	100 pg/uL	2000 pg	No
PCB CS/SS	20 uL	100 pg/uL	2000 pg	No
PAH AS	20 uL	500 pg/uL	10,000 pg	No
Injection Standard				
PCDD/F JS	20 uL	100 pg/uL	2000 pg	No
PCB JS	20 uL	100 pg/uL	2000 pg	No
PAH JS	80 uL	125 pg/uL	10,000 pg	No

Spiking profile for standard project, no split; modifications will be notated in specific sample instructions if profile changes.

16.0 Definitions

16.1 SDS – Soxhlet-Dean Stark

16.2 PPE – Personal protective equipment

16.3 XAD – Ethenyl-benzene adsorbent polymer

16.4 PCB – Polychlorinated Biphenyls

16.5 DI – Deionized (cleaned DI water by TALEX for PCBs)

16.6 GC – Gas chromatograph

16.7 SPM – Semipermeable membrane

16.8 CLLE - Continuous liquid-liquid extraction

16.9 DRBC – Delaware River Basin Commission

16.10 H_2SO_4 - Sulfuric Acid

16.11 Na_2SO_4 - Sodium Sulfate

16.12 PCDD/Fs - polychlorinated dibenzo-p-dioxin/furan

16.13 PAH – Polynuclear aromatic hydrocarbon

16.14 SPE – Solid phase extraction

16.15 P&P – Pulp and paper

16.16 Fortification – Spiking of samples with labeled standards.

16.17 Fractionation – Clean up procedures used to remove interfering compounds from an extract.

16.18 AS - Alternate Recovery Standards. A group of isotopically labeled compounds that is not otherwise designated in the analytical method for quality control (QC) purposes. Alternate recovery standards can be used to assess the recovery of a compound class relative to any step in the sampling and/or analysis procedure that is not already assessed as a mandatory part of the method, such as the cleanup step.

16.19 CS – Pre-fractionation Standard. A group of isotopically labeled compounds added in a known amount to the extract prior to cleanup.

16.20 ES – Pre-extraction Standard. A group of isotopically labeled compounds added in a known amount to the (XAD-2 adsorbent resin for air) each sample immediately before extraction and used for quantitation of target and other labeled compounds to correct for extraction, cleanup, and concentration recovery. These isotopically labeled compounds constitute a matrix spike of the resin

16.21 FS – Pre-extraction Filter Recovery Standard. A group of isotopically labeled compounds added at a known amount to the filter used to indicate the extraction efficiency of the filter media. The pre-extraction filter recovery standard is not used for quantitating or recovery correction.

16.22 JS – Pre-analysis Standard. A group of isotopically labeled compounds added at a known amount immediately prior to analysis and used to monitor instrument response, injection errors, instrument drift and to determine the recovery of the pre-extraction standard compounds.

16.23 SS – Pre-sampling Adsorbent Standard. A group of isotopically labeled compounds added in a known amount to the XAD-2 adsorbent prior to sampling used to monitor sampling aspects of the method.

16.24 TS - Pre-transport Standard. Spiking compound that can be added by the laboratory to the sample shipping containers used to transport field equipment rinse and recovery samples prior to sampling. The measured concentration of the pre-transport recovery standard provides a quality check on potential probe rinse sample spillage or mishandling after sample collection and during shipping.

17.0 References

17.1 SGS document DC_139, Appendix D Definitions.

17.2 SGS document MI_278, Waste Disposal, Pollution Prevention and Spill Clean up.

17.3 SGS document MI_141, Review of Analytical Data.

17.4 SGS document MI_46, Pipette and Syringe Calibration.

17.5 SGS document MI_13, Moisture Content

17.6 SGS document DC_353, Fortification

17.7 SGS document MI_2, Preparation of Glassware and Reusable Sample Prep Equipment.

17.8 SGS document DC_365, Fractionation

17.9 SGS document DC_379, Preparation of Fish Tissue samples for HRMS/ID analyses

17.10 SGS document MI_1, Daily Monitoring of Support Equipment

17.11 SGS document DC_364, Dioxin/Furan Analyses

17.12 EPA Method 1613B, Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, October 1994

17.13 EPA Method 1668A, Chlorinated Biphenly Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, December 1999

17.14 EPA Method 8290A, Polychlorinated Dibenzo-p-Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS), February 2007

17.15 EPA Method 1668B, Chlorinated Biphenly Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, November 2008

17.16 EPA Method 1668C, Chlorinated Biphenly Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, April 2010

17.17 SW-846 Method 3510C Revision 3, December 1996

17.18 SW-846 Method 3540C Revision 3, December 1996

17.19 SW-846 Method 3580A Revision 1, July 1992

17.20 SW-846 Method 3520C Revision 3, December 1996

17.21 Final Rule on the Methods Innovation Rule 40 CFR Parts 63, 268, et al.;

June 14th, 2005; pp. 34538-34592

17.22 40 CFR Parts 60, 63 and 266; EPA Method 23 – Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources

17.23 *Environmental Carcinogens: Methods of Analysis and Exposure Measurement Volume 11: Polychlorinated Dioxins and Dibenzofurans* (IARC Scientific Publications), C. Rappe (Editor), H. R. Buser (Editor), B. Dodet (Editor), I. K. O'Neill (Editor)

17.24 Hybrid HRGC/MS/MS Method for the Characterization of Tetrachlorinated Dibenzo-p-dioxins in Environmental Samples." Y. Tondeur, W.J. Niederhut, S.R. Missler, and J.E. Campana, *Mass Spectrom.* **14**, 449-456 (1987)

17.25 Horizon LIMS v12

17.26 APLIMS Operations Manual version 1.3.1.3

17.27 SGS template DC_560, Ultra-trace bench sheets

Standard Operating Procedure for the Preparation of Fish Tissue samples for HRMS/ID analyses

Issue date: 11/21/2022
Revision: 6

SGS North America Inc.
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Approved by:

Jeannie Milholland, Last Revised by — Date: 11-20-2022

Greg Dickinson, Technical Director — Date: 11-20-2022

Jeannie Milholland, Quality Assurance Director — Date: 11-20-2022

(Official copies of final documents will contain all three signatures.)

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Revision History

- The following change was made effective on November 21, 2022
  - Section 5 – updated temperature storage requirements
- The following change was made effective on July 8, 2022
  - Typo revision made to section 4.2 – New SOP version not required
- The following changes were made effective on June 21, 2022
  - Added section 7.6 specific to NYSDEC homogenization
- The following changes were made effective on June 21, 2022
  - Section 3 – Added requirement for cut-resistant gloves and chainmail gloves
  - Added Section 4 – Interference and Preventative Measures
  - Added Section 7.2.3 – NYSDEC homogenization requirements
  - Added Sections 7.3 & 7.4 – specific filleting requirements for NYSDEC samples
  - Section 11 – Added reference to NYSDEC SOP
- Review was completed on March 21, 2020, by Ricky Ballard and no updates needed
- The following changes were made effective on January 4, 2017
  - Section 6.3: removed references to fish fillets as this section is for whole body fish
- The following changes were made effective on May 26, 2015
  - Section 6.3.1: added 'if' before debris
  - Section 6.3.2: new section inserted, renumbering rest of section
  - Section 6.3.3: 'sample' changed to 'fillets'
  - Section 6.3.4: 'The sample is' changed to 'All fillets are' and 'together' added

1.0 Scope and Application

1.1 This standard operating procedure describes procedures for the initial preparation of fish tissue or other marine biota submitted to the laboratory which will be extracted and analyzed for ultra-trace levels of PCDD/Fs, PCBs, and/or other ultra-trace analytes by HRMS-ID technique.

1.2 This SOP is intended to provide guidance for the preliminary preparation of tissue samples prior to the sample aliquotting and analytical preparation described in individual analytical SOPs.

1.3 Customer specific QAPP/SAP/SOW/protocols with alternate procedures will take precedence over this SOP.

2.0 Summary of Method

2.1 Fish tissue samples are inherently nonhomogeneous and require special treatment to obtain a representative sample for analysis. This SOP provides guidance on appropriate procedures to follow to obtain a representative sample for extraction and analysis for samples analyzed by HRMS-ID methods.

3.0 Safety Precautions

3.1 Gloves must be worn when handling samples, standards, and reagents.

3.2 Safety glasses must be worn at all times when working in the laboratory.

3.3 A lab coat must be worn while dealing with samples, standards, and reagents.

3.4 Cut gloves are to be worn when filleting fish using a knife. Chainmail gloves are to be worn when using a cleaver.

3.5 Avoid distractions when working with sharp equipment to minimize the loss of focus to the task. Do not place any spoons, spatulas, knives, or any other devices into a food processor when in operation. Be certain the unit has stopped rotating BEFORE opening the lid.

3.6 In the interest of safety and pollution prevention, all spills must be cleaned up immediately.

3.7 Waste is disposed of in appropriate waste containers in accordance with the local, state and federal regulations. No sample or reagent is ever disposed of down a drain or in the trash. See document MI_278 for waste disposal, pollution prevention and spill cleanup.

4.0 Interference and Preventative Measures

4.1 Conduct all work in a clean laboratory environment. All worksurfaces, utensils and grinder work bowls and covers should be cleaned with soap and water, then rinsed with clean water, prior to working with samples, between each sample, and upon completion of sample preparation for the day. Alternatively, between samples aluminum foil may be placed on the work surface for the succeeding fish sample; discard foil after one use.

4.2 Wear nitrile or latex gloves at all times while preparing samples. Dispose of gloves between samples and place new gloves on hands. If a glove is torn or punctured, immediately discard the glove and replace with a new glove. Discard gloves at the end of the day, or earlier if they become unsuitable for clean preparation of samples.

4.3 Rinse fish samples in clean DI water if soil, debris or other matter are evident on the exterior surfaces. Allow water to run off and dry exterior surface.

4.4 Following preparation of sample portions, place sample in clean containers of suitable size for the sample. Place small samples in chemically clean glass jars, cover, and label immediately. Jars should have PTFE-lined caps and be recleaned and certified. For large samples, wrap in Acetone or methanol rinsed aluminum foil and label externally. For NYSDEC samples rinse the aluminum foil with Hexane. Place foil wrapped sample in a labeled food-grade plastic bag for subsequent storage and transport. If hexane-rinsed aluminum foil is unavailable, and samples are not to be analyzed for phthalates, the excised sample may be placed in a food grade plastic bag, labeled externally and placed in frozen storage.

5.0 Sample Preservation and Storage

5.1 Fish samples are kept frozen at ≤-10°C until ready for extraction in which they are placed in refrigeration at 0-6°C) to thaw for 2 days.

5.2 After extraction, the samples are placed in the freezer and kept frozen until disposal.

6.0 Equipment and Supplies

6.1 Hobart food chopper, or comparable device (blades must be sharp)

6.2 Tissuemizer (blades must be sharp)

6.3 Waring blender, or similar device (blades must be sharp)

6.4 Glass or PTFE cutting boards

6.5 Knives and cutting implements, stainless steel (blades must be sharp)

6.6 Standard laboratory glassware, amber glass jars/bottles

6.7 Heavy duty aluminum foil

6.8 Pesticide grade or better, acetone and/or methanol

6.9 Balance capable of weighing to 0.1 g

6.10 Blade sharpener

7.0 Sample Preparation

7.1 Cleaning

7.1.1 Prior to handling samples, any utensils, knives, cutting boards and bench surfaces should be thoroughly cleaned and rinsed/wiped with acetone or methanol.

7.1.2 Implements should be washed in detergent hot water solution, rinsed with tap water, followed by rinsing with DI water, and acetone or methanol before use and in between each individual sample that is prepared.

7.1.3 Pre-cleaned certified amber glass jars or bottles may be used without any further cleaning.

7.1.4 Heavy duty aluminum foil should be rinsed with acetone or methanol prior to use or before contact with any sample.

7.2 Fish Fillets

7.2.1 Fish samples often arrive at the lab having already been filleted and packaged by the client. Instructions from the client and/or project manager should be followed for appropriate compositing.

7.2.2 Samples may be ground and homogenized using the Hobart or Waring blender, depending on the size of the sample(s).

7.2.3 For NYSDEC samples Mix the tissue and repeat the grinding step at least two more times and until the sample appears to be homogeneous.

7.2.4 Samples should be kept cold. Partially frozen tissue is typically more readily homogenized.

7.2.5 Individual (single) fillets are ground and homogenized to fine paste-like consistency to ensure that the sample is thoroughly homogenized and mixed.

7.2.5.1 The entire fillet received should be homogenized. Large fillets may be cut with a knife into smaller pieces to facilitate homogenization.

7.2.5.2 If enough homogenized sample is available, the sample should be placed into three labeled 4-ounce amber jars then moved to appropriate cold storage.

7.2.5.3 Any remaining sample may be discarded, unless directed otherwise.

7.2.6 Composited/multiple fillets are ground and homogenized to fine paste-like consistency to ensure that the sample is thoroughly homogenized and mixed.

7.2.6.1 Whole fillets received should be used and not sub-sampled prior to homogenization.

7.2.6.2 Large fillets may be cut with a knife into smaller pieces to facilitate homogenization.

7.2.6.3 Component fillets may be homogenized together if the fillets are small enough to fit into the Hobart or Waring blender. Care must be taken to ensure that component fillets are thoroughly homogenized and mixed to provide a representative composited sample.

7.2.6.4 If enough of the sample is available, the composited sample should be placed into three labeled 4-ounce amber jars then moved to appropriate cold storage.

7.2.6.5 Any remaining sample may be discarded, unless directed otherwise.

7.3 Standard Fillet for NYSDEC samples

7.3.1 Remove scales from the fish making sure not to remove the skin.

7.3.2 Take the fillet from the left side of the fish

7.3.3 Make a cut along the ventral midline of the fish from the vent to the base of the jaw.

7.3.4 Make a diagonal cut from the base of the cranium following just behind the gill to the ventral side just behind the pectoral fin.

7.3.5 Remove the flesh and ribcage from the left side of the fish by cutting from the cranium along the spine and dorsal rays to the caudal fin. The ribs should remain on the fillet.

7.3.6 Score the skin and homogenize the entire fillet.

7.4 Modifications to the Standard Fillet for NYSDEC samples

7.4.1 Four modifications of the standard fillet procedure above are designed to account for variations in fish size or known

preferred preparation methods of the fish for human consumption.

7.4.2 Some fish are too small to fillet by the above procedure. Fish less than approximately 6 inches long and rainbow smelt are analyzed by descaling, cutting the head off from behind the pectoral fin and eviscerating the fish. Ensure that the belly flap is retained on the carcass to be analyzed.

7.4.3 Some species are generally eaten by skinning the fish. The skin from these species is also relatively difficult to homogenize in the sample. Hence, for the following list of species, the fish is first skinned prior to homogenization: Brown Bullhead, Yellow Bullhead, Black Bullhead, Atlantic Sturgeon, White Catfish, Channel Catfish and Lake Sturgeon. Note: For the catfish and bullhead it can be easier to remove the skin after filleting. Either before or after filleting is fine as long as muscle is not lost in the removal of the skin.

7.4.4 American eel are analyzed by removing the head, skin, and viscera; filleting is not attempted.

7.4.5 Forage fish and young-of-year fish are analyzed whole. Small prey fish should have been composited prior to delivery to the laboratory. Individuals should be of the same species and size matched, where the smallest fish is at least 75% of the largest in length.

7.5 Whole body fish

7.5.1 Partially frozen whole body fish samples should be rinsed with DI water if debris or mucus are visible.

7.5.2 All whole body fish are thoroughly ground and homogenized together in the Hobart or Waring blender. Fish skin may be difficult to thoroughly homogenize. Care will be taken to homogenize the sample to the extent possible with the ideal final consistency being paste-like.

7.5.3 The sample should be placed into three labeled 4-ounce amber jars then moved to appropriate cold storage.

7.5.4 Any remaining sample may be discarded, unless directed otherwise.

7.6 NYSDEC Fish Homogenate criteria

7.6.1 Sample must be uniform.

7.6.2 No whole fish or fish organs may be present.

7.6.3 All sport fish must be descaled including those under 6 inches in length.

7.6.3.1 Sport fish fillets (e.g., smallmouth bass, walleye, pumpkinseed), small sport fish with head and viscera removed, and small prey (forage) fish should be thoroughly mixed, with only occasional small pieces of skin or bone.

7.6.3.2 Three or fewer pieces less than 1 cm of skin or bone may be present.

7.6.4 Carp – Four to Six or fewer pieces less than 2-3 cm of skin or bone may be present.

7.6.5 Large prey fish – are expected to have some larger pieces of skin or bone (3 – 6 cm) but must be evenly distributed; occasional occurrence of fins is also acceptable.

8.0 Quality Control Requirements

8.1 Data outside of QC limits may be addressed by one or more of the following options:

8.1.1 Re-preparation and re-analysis of sample

8.1.2 Client notification

8.1.3 Discussion and qualification of data by case narrative

8.1.4 Re-sampling and re-analysis (client decision)

8.2 Data outside of QC limits may be reported if directed by the client. It must be qualified by a case narrative detailing the QC problems with advice on the usability of the data.

8.3 SGS personnel performing this procedure must have documented demonstration of capability and must have signed attestation for current revision of this SOP.

9.0 Data Review and Reporting Requirements

9.1 This section addresses any criteria that must be met before the analytical results can be reported and should also reference our Data Review SOP (MI_141).

10.0 Definitions

10.1 QAPP – Quality Assurance Project Plan

10.2 SAP – Sampling and Analysis Plan

10.3 SOW – Scope of Work

10.4 SOP – Standard Operating Procedure

10.5 PTFE – Polytetrafluoroethylene (Teflon)

10.6 PCDD/F - Polychlorinated dibenzo-p-dioxin/furan

10.7 PCB - Polychlorinated biphenyl

10.8 HRMS-ID – High-resolution Mass Spectrometry Isotope Dilution

11.0 References

11.1 Prep Lab Standard Operating Procedure NYS Department of Environmental Conservation, Hale Creek Field Station ; SOP PrepLab-4 (5-28-2014)

11.2 SGS document DC_139, Appendix D Definitions.

11.3 SGS document MI_278, Waste Disposal, Pollution Prevention and Spill Clean up.

11.4 SGS document MI_141, Review of Analytical Data.

Standard Operating Procedure for the Extraction of Polychlorinated Biphenyls (PCBs as Aroclors) in Solids by Soxhlet Extraction: Method 3540C

Issue date: 11/17/2022
Revision: 1

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Approved by:

Antonino Hernandez, Last Revised by — Date: 11/17/22

Greg Dickinson, Technical Director — Date: 11-17-2022

Jeannie Milholland, Quality Assurance Director — Date: 11-17-2022

(Official copies of final documents will contain all three signatures.)

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Revision History

- The following changes were made effective on 11/17/2022:
  - Sections 5.1, 5.3; updated temperatures
  - Section 9.1.1; added reference
  - Section 9.1.2; removed section
  - Sections 9.1.10, 9.1.11, 9.1.12; updated wording and added sample containers
- The following changes were made effective on 2/9/2022:
  - New SOP

1.0 Scope and application

1.1 This method details procedures used in the extraction of solid materials (e.g., soils, sediments, and solid waste) for the analysis of polychlorinated biphenyls (PCBs).

1.2 This SOP is based on SW-846 Method 3540C.

1.3 See Document DC_139 Appendix D for a full list of definitions.

2.0 Summary of method

2.1 Approximately 30 g of solid are mixed with anhydrous sodium sulfate and transfer into an extraction thimble. Samples are extracted with Hexane using a Soxhlet extractor apparatus to extract analyte of interest. The extract is then dried and concentrated. Finally, extract is brought to volume, and analyzed by injection on a GC with an ECD detector according to method 8082A.

3.0 Safety precautions

3.1 All preparations will be performed in a fume hood. This method measures compounds that can evaporate into the air. The analyst must use the fume hood to sweep any evaporated compounds out of the breathing zone. Some target analytes for this method are known or suspected health risks.

3.2 Gloves must be worn when handling samples, standards, and reagents.

3.3 Safety glasses must be always worn when working in the laboratory.

3.4 A lab coat must be worn while dealing with samples, standards, and reagents.

3.5 In the interest of safety and pollution prevention, all spills must be cleaned up immediately.

4.0 Interferences and preventative measures

4.1 Method interferences may be caused by contaminants in solvents or reagents, out-gassing from equipment plumbing, the diffusion of compounds through the bottle seal during storage and handling, and laboratory solvent vapors. This can lead to discrete artifacts and or elevated baseline in the gas chromatograph. All these materials must be demonstrated to be free from interferences by analyzing laboratory reagent blanks.

4.2 All glassware and some solid reagents must be scrupulously cleaned to

remove interferences from previous extractions and from phthalates. These materials are washed before each use and baked in a muffle furnace at 400°C to remove these interferences.

4.3 Pesticide grade solvents (or equivalent) shall be used for standards and extractions.

5.0 Sample collection, preservation, and storage

5.1 Samples must be protected from light and iced or refrigerated at 0 - 6°C from the time of collection until extraction. Temperature control of the samples from collection until laboratory receipt is beyond laboratory control. However, the temperature of the samples is recorded upon receipt and inspection. Any discrepancies are noted.

5.2 All samples must be extracted within 1 year of sample collection.

5.3 Extracts must be stored at 0-6°C.

5.4 Extracts must be analyzed within 40 days of extraction.

5.5 Solid samples must be collected in amber glass containers with a Teflon coated top. The jars must be chilled after being filled and protected from light.

5.6 All laboratory samples and waste are stored and disposed of in accordance with state and federal regulations.

5.7 Waste is disposed of in appropriate waste containers in accordance with the local, state, and federal regulations. No sample or reagent is ever disposed of down a drain or in the trash. See document MI_278 for waste disposal, pollution prevention and spill cleanup.

6.0 Equipment and supplies

6.1 Soxhlet setup

6.1.1 Soxhlet body equipped with drain tube and

6.1.2 Dean Stark

6.1.3 Condenser

6.1.4 Glass thimble (glass fritted at bottom)

6.1.5 500mL round bottom flask

6.2 Suitable personal protection equipment (PPE)

6.3 Aluminum foil

6.4 Teflon Boiling chips

6.5 Spatulas

6.6 60 mL glass vials with Teflon coated closures.

6.7 Pipettes

6.8 Analytical Balance

6.9 Drying Oven

6.10 Desiccator.

6.11 Aluminum weight boats

6.12 Heating mantle

6.13 Chiller

6.14 Glass Beakers (various sizes)

6.15 4 or 8 oz. jars

6.16 Glass funnels

6.17 40- or 60-ml vials

6.18 Fume hood TurboVap

6.19 Diatomaceous earth (Hydromatrix)

7.0 Standards and reagents

7.1 Acetone – Pesticide Grade

7.2 Hexane – Pesticide Grade

7.3 Anhydrous Sodium sulfate - Baked at 400°C for 4 hours and stored in a labelled glass container.

7.4 Sulfuric acid, concentrated

7.5 Surrogate Standard – for all analyses, use 1 mL of stock surrogate standard per extraction vessel. See the PCB method SOP for the exact composition and concentration of this standard.

7.6 LCS Spiking solution- for all analyses, use 1 mL of stock LCS standard per extraction vessel. See the PCB method SOP for the exact composition and concentration of this standard.

7.7 MS/MSD Spiking solution– for all analyses, use 1 mL of stock MS spiking standard per extraction vessel. See the PCB method SOP for the exact composition and concentration of this standard.

7.8 See Table 1 for Spiking Profiles for all LCS, MS/MSD solutions and surrogate compounds

8.0 Calibration

8.1 Not applicable

9.0 Sample preparation

9.1 Sample Handling

9.1.1 Sediments/soil samples: Inspect sediments sample for any aqueous layer and, if presents, discard water layer. Thoroughly mix samples and discard any foreign objects (sticks, leaves, rocks, & shells) not representative of the subsampling.

9.1.2 Determination of percent solid: See SOP MI_13

9.1.3 All QC (MB,LCS,LCSD) samples contains anhydrous sodium sulfate or Hydromatrix as a matrix.

9.1.4 Place a glass container (8oz jar or 250mL beaker) and place on balance. Tare out the container weight and add approximately 30g of samples.

9.1.5 Add appropriate amount of anhydrous sodium sulfate and homogenize together. If samples contain high amount of moisture use Hydromatrix to absorb the liquid from the sample.

9.1.6 Transfer mixture into an extraction thimble and place in a Soxhlet apparatus setup. Use a Dean Stark trap when large amounts of moisture are present

9.1.7 Obtain a 500mL round bottom flask and add approximately 300mL of Hexane with some boiling chips.

9.1.8 See Table 1 in Section 15 for amounts to be spiked.

9.1.9 Attach Soxhlet to the round bottom and extract samples for 16-24hrs at 4-6 refluxes cycle/hour.

9.1.10 Concentrate the sample after the extraction period by opening the stop cock and draining out solvent to waste.

9.1.11 Allow the sample to concentrate down to approximately 20-30 mL in the round bottom on the heating mantle. Dry the extract if water is visible by adding small amount of sodium sulfate to the round bottom.

9.1.12 Transfer the extract to a 60 mL vial and collect any rinses.

9.1.13 Place the 60 mL vial in the TurboVap and blow down the extract using N_2 at about 5-10psi and at a temperature of 40-50°C to approximately a 10 mL final volume. A reference vial will be used to determine 10 mL final volume.

9.1.14 Samples are cleaned up with a Sulfuric Acid technique. Refer to SV_363.1 (Sulfuric Acid Cleanup-M3665A). Additional cleanup maybe required for high sulfur residue using copper powder refer to SV_138.5 (3660 Sulfur Cleanup).

9.1.15 Store 1 mL of the extract in Teflon lined silicone septa open-top screw-cap GC vials until ready for analysis.

10.0 Analytical procedure

10.1 Please see SOP SV_70- Determination of Polychlorinated Biphenyls (PCBs as Aroclors)

11.0 Details of calculations

11.1 % dry weight = g of dry sample/ g of sample ×100

12.0 Quality control requirements

12.1 The following items are general requirements. See the SOP of the associated determinative method for specific requirements.

12.2 A batch is composed of twenty samples. At a minimum, each batch has a method blank, LCS, and MS/MSD. All quality control samples must be prepared in the same way as samples.

12.3 Data outside of QC limits may be addressed by one or more of the following options:

12.3.1 Re-preparation and re-analysis of sample

12.3.2 Client notification

12.3.3 Discussion and qualification of data by case narrative

12.3.4 Re-sampling and reanalysis (client decision)

12.4 Data outside of QC limits may be reported if directed by the client. It must be qualified by a case narrative detailing the QC problems with advice on the usability of the data.

13.0 Data review and reporting requirements

13.1 See section 13 of the PCB method SOP SV_70.

13.2 Refer to SGS document MI_141, Data review SOP

14.0 Preventative maintenance

14.1 All instrumentation is serviced by an external instrumentation service vendor or by SGS personnel trained in preventative maintenance.

Preventative maintenance is performed as needed on all equipment. All instrument preventative maintenance is performed according to the manufacturers recommended procedures. All maintenance shall be thoroughly documented in the instrument maintenance log.

15.0 Tables

Table 1: Spiking Profiles for all LCS, MS/MSD solutions and surrogate compounds

8082 Soil	Volume (µL)	[Conc] (µg/mL)	Amt. (µg)	Final Vol. (mL)	[Ext/Inst] (µg/mL)
Surrogate	1000	1	1	10	0.1
Matrix Spike(LCS)	1000	10	10	10	1
Matrix Spike(MSD)	1000	10	10	10	1

16.0 Definitions

16.1 PQL – Practical quantitation limit

16.2 LCS – Laboratory control spike

16.3 LCSD - Laboratory control spike duplicate MS – Matrix spike

16.4 MSD – Matrix spike duplicate

16.5 PCBs - polychlorinated biphenyls

16.6 GC - gas chromatograph

16.7 ECD - electron capture detector

16.8 SOP - standard operating procedure

16.9 QC - Quality control

17.0 References

17.1 SW-846 Method 3540C Revision 3, December 1996

17.2 SGS document DC_139, Appendix D Definitions.

17.3 SGS document MI_278, Waste Disposal, Pollution Prevention and Spill Clean up.

17.4 SGS document MI_141, Review of Analytical Data.

17.5 SGS document SV_363.1 (Sulfuric Acid Cleanup-M3665A).

17.6 SGS document SV_138.5 (3660 Sulfur Cleanup)

Standard Operating Procedure for the Determination of Lipids in Tissue

Issue date: 6/14/2023
Revision: 2

SGS North America Inc.
5500 Business Drive
Wilmington, North Carolina 28405

Approved by:

J. Harrison Land, Last Revised by — Date: 6/14/2023

Greg Dickinson, Technical Director — Date: 6-14-2023

Jeannie Milholland, Quality Assurance Director — Date: 6-14-2023

(Official copies of final documents will contain all three signatures.)

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Revision History

- The following changes were made effective on June 7, 2023:
  - Updated TOC
  - Section 2.0 revised equation
  - Section 7.7 updated reference for NYSDEC
  - Section 7.9 added details
  - Section 7.10 added details
- The following changes were made effective on April 5, 2023:
  - Section 2 added equation
  - Section 4 added information about holding times
  - Section 5 added desiccator, fritted funnel, rotary evaporator and TurboVap, update weight of Al weigh boats
  - Section 7 added desiccator and drying times to procedure, reference to DC_560 and other details
  - Section 8 added approximate weight of aluminum pans and revised equation
  - Section 9 – new section for definitions
  - Section 10 added reference to DC_560 and 3550C.
- The following changes were made effective on February 2, 2022:
  - Section 2.1 Added reporting limit information
  - Section 5.0 Added balance details
  - Section 7.0 updated procedure with additional details
- The following changes were made effective on February 2, 2022:
  - Added Sections 5.6 – 5.8 to Equipment and Supplies
  - Added Section 6 (Reagents and Standards)
  - Replaced "MC" with methylene chloride throughout the SOP
- SOP has been reviewed by Amber Nunalee, on 7/2/2020 and no revisions are necessary
- Review was completed on March 21, 2020, by Ricky Ballard and no updates needed
- The following changes were made effective on August 12, 2015:
  - New SOP converted to SGS format from AP-SP-C3

1.0 Scope and Application

1.1 To describe the procedures followed for the determination of lipids in tissue samples

1.2 See Document DC_139 Appendix D for a full list of definitions.

2.0 Summary of Method

2.1 An aliquot of the homogenized samples is weighed out separately and sonicated. Reporting Limit of 0.05% is achieved using 8 grams of sample on a balance capable of 0.00001 g resolution per the following calculation.

*Wt. diff =0.1% margin=> Uncertainty = 0.001*2(Uncertainty of W_1 and W_2) => 0.002g/8g (sample wt.) = 0.00025g*100 = 0.025% * (2 crit wt.) = 0.05% accuracy with certainty*

3.0 Safety Precautions

3.1 Gloves must be worn when handling samples, standards, and reagents.

3.2 Safety glasses must be always worn when working in the laboratory.

3.3 A lab coat must be worn while dealing with samples, standards, and reagents.

3.4 In the interest of safety and pollution prevention, all spills must be cleaned up immediately.

4.0 Sample Collection, Preservation and Storage

4.1 Tissue samples are kept frozen until thawed for extraction.

4.2 Holding times begin and are calculated when the fish is fully thawed.

4.3 Waste is disposed of in appropriate waste containers in accordance with the local, state, and federal regulations. No sample or reagent is ever disposed of down a drain or in the trash. See document MI_278 for waste disposal, pollution prevention and spill cleanup.

5.0 Equipment and Supplies

5.1 Meat grinder/Blender

5.2 Freezer

5.3 Glass container (40 mL vials, 60mL vials or 4 oz jars)

5.4 Balance capable of Resolution to 0.00001 grams

5.5 Aluminum foil

5.6 Forceps

5.7 Sonicator

5.8 ~ 1 gram Aluminum weigh boats

5.9 Desiccator

5.10 Fritted funnel

5.11 Rotary Evaporator

5.12 Turbovap

6.0 Standards and Reagents

6.1 Acetone

6.2 Methylene Chloride

6.3 Sodium Sulfate

7.0 Procedure

7.1 Weigh and record approximately 8 g of the ground and homogenized tissue sample into a tarred container appropriate for sonication extraction on a balance capable of resolving weight to 0.00001g.

7.2 Add 20 mL of 1:1 methylene chloride: acetone and sonicate for 15 minutes. Let sit in hood overnight with cap on. Tissue must be completely submerged in the solvent.

7.3 Transfer solvent to appropriate glass container carefully so as not to transfer any tissue, add 20 ml of methylene chloride to the original extraction container, and shake for 1 minute.

7.4 Repeat step 7.3., for a total of 60mL of extraction solvent.

7.5 Pass solvent through a Na2SO4 plug into a 4-oz jar or other appropriate container (round bottom flask for Roto-vap/ Glass borosilicate vial for TurboVap.)

7.6 Make sure that all water has been removed and no Na2SO4 crystals are present. If crystals are present repeat step 7.5

7.7 Concentrate the extracted sample using the rotary evaporator, TurboVap, or ambiently in a clean hood. (For NYSDEC samples TurboVap will be the primary form of sample concentration, with Roto-vap as a backup if

significant amounts of solvent are required)

7.8 Pre-weigh an aluminum boat on the analytical balance to the nearest 0.00001 g. Use forceps when handling the aluminum boat and do not touch the boat with fingers.

7.9 Transfer the lipids from the extraction vessel to the aluminum boat using a small amount of DCM (~3-4mL) to ensure quantitative transfer of lipids, allow the transfer solvent to evaporate in the fume hood (minimum time of 30 minutes), and place the sample inside the desiccator for thirty minutes to ensure dryness. Weigh to the nearest 0.00001 g.

7.10 Repeat cycle of evaporation to dryness for an additional 30 minutes until the mass change of the residue is less than 1.0 % of the previous weight to ensure the solvent has been completely removed.

7.11 Record weight on SGS % Lipids Determination worksheet DC_560

8.0 Details of Calculations

8.1 As per the method, calculate the lipids content to the nearest three significant figures using the formulae:

8.2 Lipid/Oil residue weight

Residue wt. = (Tare wt. with dried sample) – Tare wt.

8.3 Oil/Lipid Content:

$$\%Oil/Lipid = \left(\frac{Residue\ Weight}{Sample\ Weight}\right) \times 100$$

where,

Residue wt. = final weight after drying
Sample wt. = initial weight of sample
tare = weight of pan (should be approximately 0.8 grams)

9.0 Definitions

9.1 Desiccator – an apparatus that hold a drying agent for removing moisture from specimens and protecting specimens from water vapor in the air.

9.2 NYSDEC – New York State Department of Environmental Conservation

10.0 References

10.1 SGS document DC_139, Appendix D Definitions.

10.2 SGS document MI_278, Waste Disposal, Pollution Prevention and Spill Clean up.

10.3 SGS document DC_560, % Lipid Determination Worksheet

10.4 SW-846 Method 3550C : Ultrasonic Extraction Revision 3 February 2007

10.5 SGS document DC_560, Bench Sheet (Including % Lipid)

Standard Operating Procedure for the Determination of Polychlorinated Biphenyls (PCBs as Aroclors) By Gas Chromatography SW-846 8082A and EPA 608.3

Issue date: 11/17/2022
Revision: 27

SGS North America Inc.
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Approved by:

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Antonino Hernandez, Last Revised by — Date

Greg Dickinson — 11-17-2022
Greg Dickinson, Technical Director — Date

Jeannie Milholland — 11-17-2022
Jeannie Milholland, Quality Assurance Director — Date

(Official copies of final documents will contain all three signatures.)

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Revision History

- The following changes were made effective on November 17. 2022:
  - Section 5.2, 5.11.2.2.2, 7.9, table 13; updated temperature
- The following changes were made effective on January 18, 2022:
  - Section 1.1, 2.1; updated matrix
  - Section 3.4, 4.7, 5.5, 5.11.1.2, 5.12.3, 5.12.6; sections added
  - Section 4.3, 9.1, 9.2, 12.3.1, 13.5; updated section
  - Section 5.3; updated holding time
  - Section 6.0; updated equipment and supplies
  - Section 15.0, Table 8, 9 and 13; updated table with soil matrix
  - Section 17.0; updated references
- The following changes were made effective on May 5, 2020:
  - Section 3.2.3; added Chemwatch for MSDSs and updated PPE
  - Section 8.1.5; added new to calibration
  - Section 12.9.2; updated to excel spreadsheet
  - Section 13.2.1.2; removed section
- The following changes were made effective on May 10, 2018:
  - Updated Table 9 to reference method 8082A
- The following changes were made effective on April 20, 2018.
  - Sections 15.1, 15.8, 15.9, 15.13; updated tables
  - Section 17.11; updated reference
- For revisions made effective on February 15, 2018 or before, see earlier version of SOP

1.0 Scope and Application

1.1 This method may be used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors in extracts from solid (typically soils and sediment), aqueous matrices, using open-tubular, capillary columns with electron capture detectors (ECD). Specifically, it may be used to detect the PCB Aroclors listed in Table 1.

2.0 Summary of Method

2.1 This method contains chromatographic conditions for the detection of PCBs. Prior to the use of this method, appropriate extraction and clean up techniques must be performed. Groundwater and other aqueous samples are extracted with methylene chloride using continuous liquid-liquid extraction (method SW3520). Solid samples are extracted with hexane by manual Soxhlet (method SW3540). A 1.0 µl sample is auto injected into the gas chromatograph and the halogenated components are detected by an electron capture detector (ECD). All target hits are qualitatively confirmed by analysis on a second GC column.

3.0 Safety Precautions

3.1 Because of the toxicity of many of these compounds, the analyst must take the necessary precautions to prevent exposure to materials known or believed to contain PCBs. It is the responsibility of the laboratory personnel to ensure that safe handling procedures are employed.

3.2 Solvent Spills

3.2.1 Immediately alert others in the affected area and notify the appropriate supervisor. If necessary, evacuate the area.

3.2.2 Attend to any person(s) who may be contaminated. Contaminated clothing must be removed immediately, and any affected skin flushed with water.

3.2.3 Don PPE (personal protective equipment) as appropriate to the spill. MSDSs are may be found in Chemwatch reference the appropriate gloves and PPE to be used for handling each chemical.

3.2.4 Within each spill cart are spill socks/booms and spill pillows. Use the socks/booms to contain the spill and prevent further spread of the solvent. Spill pillows can be used to absorb the remaining solvent.

3.2.5 Used sorbant materials are to be placed in the provided

hazardous waste material bags and disposed of accordingly.

3.2.6 If a volatile solvent is spilled, ventilate the area as much as possible, i.e. open hoods, open rear doors, and if necessary, use portable fans.

3.3 Acid and Caustic Spills

3.3.1 Attend to any person(s) who may be contaminated. Contaminated clothing must be removed immediately, and any affected skin flushed with water.

3.3.2 Don PPE (personal protective equipment) as appropriate to the spill.

3.3.3 Contain the spill if required using spill socks or spill pillows.

3.3.4 Within each spill kit are acid and caustic neutralizer chemicals. Additional acid neutralizer is also stored in the shipping/receiving area.

3.3.5 Distribute the neutralizers evenly over the spilled liquid. Using the included scoop, work from the outside of the spill towards the center. This technique prevents splashing and any further spread of the liquids.

3.3.6 When the spilled materials have been absorbed, use the brush and scoop to place materials in an appropriately labeled hazardous waste material bag.

3.3.7 Neutralized acid and caustic waste can be disposed into the regular trash.

3.3.8 Decontaminate surfaces involved using mild soap and water, as appropriate.

3.4 Non-Indigenous Soil Spill

3.4.1 Locate several containers, such as 4 oz. jars, which will be used to package the spill.

3.4.2 Label the jars with the purple stickers that indicate that the contents are non-indigenous.

3.4.3 Anything that comes into contact with the soil must then be stored in the waste jars.

3.4.4 Use paper napkins, aluminum foil, and the like to clean up small spills.

3.4.5 If a spill warrants the use of a whisk broom and dust-pan then they must be labeled with the sticker to ensure proper disposal.

3.5 Each employee must read, understand and follow the safety guidelines in the Chemical Hygiene Plan, SGS document MI_254.

3.6 For further safety precautions, please refer to the referenced test method.

4.0 Interferences and Preventative Measures

4.1 Refer to Method 8000B.

4.2 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks.

4.3 Interferences co-extracted from the samples will vary considerably from sample to sample. While general clean up techniques are performed during the extraction process, unique samples may require additional clean up. Single-component chlorinated pesticides such as the DDT analogs (DDT, DDE, and DDD) may trigger the detector to respond.

NOTE: As a general practice a standard of DDT analogs should be injected to determine if any Aroclor peaks may be subject to interferences with the column in use. Some soil and sediment samples may have considerable DDT interference with Aroclor 1254 last major peak.

4.4 Clean all glassware prior to use with soap and water, rinse with DI water. Glassware is then fired in a muffle furnace.

4.5 Interferences by phthalate esters may pose a problem in the detection of PCBs. Minimizing contact with any type of plastic material will help to avoid this interference.

4.6 Contamination by carry over can occur whenever high-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank to check for cross contamination.

4.7 Sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences. Sulfur should be expected from sediment samples as well. The removal of sulfur should use Method 3660.

5.0 Sample Collection, Preservation, and Storage

5.1 See the introductory material to Method 8000B, Organic Analytes, Sec. 4.1. A one-liter sample should be collected in a glass-amber bottle.

5.2 All samples for PCB analysis must be protected from light and

refrigerated at 0-6°C from the time of receipt (time of collection when possible) until the time of extraction.

5.3 Every effort is always made to extract samples within holding time. All water samples for semi-volatile analysis shall be extracted within 7 days time of sampling, or less. All soil and sludge samples for semi-volatile analysis shall be extracted within 14 days of sampling. All extracts are analyzed within 40 days from the time of extraction.

5.4 Water samples may be collected in amber glass containers having a total volume of at least 1L with a Teflon-lined screw-top cap. Deviations from the specified container must be noted on the sample receipt checklist.

5.5 Soil samples may be collected in amber glass wide-mouth containers having a final weight of at least 4 ounces with Teflon lined screw-top lid. Deviations from the specified container must be noted on the sample receipt checklist.

5.6 SGS offers pre-cleaned and preserved sample containers for this method.

5.7 Samples are retained for 30 days after analysis unless the client makes other arrangements. Samples are then disposed in waste containers for removal by a disposal contractor. Sample aliquots are never poured down the drain or placed in the sanitary trash system. Pollution control is the responsibility of all personnel handling samples.

5.8 Wastes from various processes within the laboratory must be separated based on type (solid, solvent, waste oil, etc.) and then disposed of properly. Non-hazardous items such as solvent-rinsed, air-dried glass vials may be disposed of in the trash. Solvents are stored in drums and disposed of through a waste disposal company. Other solid waste is disposed of through a waste disposal company.

5.9 Individuals working in the lab must be familiar with the properties and hazards of the materials with which they work. In the event of a chemical spill, it is the responsibility of the individual(s) for prompt and proper clean-up of the spill. Before beginning work with chemicals, all employees must be familiar with the proper techniques for spill clean up, and the location of readily available appropriate clean-up materials.

5.10 All laboratory waste is stored and disposed of in accordance with state and federal regulations.

5.11 Disposal of used laboratory supplies

5.11.1 Prep area waste

5.11.1.1 Water Extraction Waste

5.11.1.1.1 Solvent waste generated during the cleanup of samples is collected in a fluorinated carboy.

This carboy is then emptied into the solvent waste drums on a periodic schedule or as sample workload mandates.

5.11.1.1.2 Gloves, pipettes, paper towels and other disposable lab products go in the trash.

5.11.1.1.3 Solvent-rinsed sample vials and collection vials are air dried and disposed of in the trash.

5.11.1.1.4 Used media is collected in a central container and dried in the vacuum hood. When the solvent saturated media is completely dried, it is disposed of in the trash.

5.11.1.1.5 Solvent used for hardware cleaning is dumped into the solvent waste drum.

5.11.1.2 Soil Extraction Waste

5.11.1.2.1 All waste solvent is collected and then disposed of in the solvent waste drum.

5.11.1.2.2 Soil that has been extracted is disposed of with lab trash.

5.11.1.3 Glassware Cleaning

5.11.1.3.1 All dishwater is poured down the sink. Copious amounts of water are flushed down the sink along with the dishwater.

5.11.1.4 Turbovap

5.11.1.4.1 Solvent saturated paper towels are disposed of with lab trash.

5.11.2 Analytical instrument area waste

5.11.2.1 Solvent Waste

5.11.2.1.1 Rinse vial waste from the autosamplers is collected and disposed of in the solvent waste drum.

5.11.2.2 Sample extract vials

5.11.2.2.1 All vials are collected and archived.

5.11.2.2.2 Extracts must be stored at 0-6˚C.

5.11.2.2.3 Extracts must be analyzed within 40 days of extraction. After the holding times have

expired, extract vials are placed in a waste drum for disposal.

5.11.2.3 Standard Vials

5.11.2.3.1 All standard vials are disposed of with extract vials.

5.12 Waste Removal

5.12.1 Sample Tracking

5.12.1.1 All analyzed samples are removed from the storage cooler ~30 days after the final report has been delivered to the client. This time may vary based on specific client contract requests. Pulled samples are maintained on storage shelves an additional 30 days prior to disposal.

5.12.2 Solvent Waste

5.12.2.1 All solvents are collected in a labeled 55-gallon drums. Each drum is also labeled with start and end dates.

5.12.3 Solid Waste

5.12.3.1 All solid samples are ground into 55-gallon drums labeled for solids only.

5.12.4 Hazardous Waste

5.12.4.1 High-level PCB that are greater than 50ppm are segregated and disposed of in the hazardous waste drum or returned to the client. All returned samples are shipped following DOT shipping guidelines.

5.12.5 Aqueous Waste

5.12.5.1 All aqueous samples are disposed of into polypropylene lined 55-gallon drums labeled for aqueous samples only.

5.12.6 Non-Indigenous Waste

5.12.6.1 Having been identified at login and labeled with brightly colored “non-standard waste” stickers, non-indigenous solid samples are disposed of by heating in the oven for at least 2 hours at 150°C. They are cooled and disposed of in the 55-gallon drum labeled for solids only. Non-indigenous aqueous samples are collected into the aqueous waste drums for incineration.

5.13 When enough drums have accumulated in six months or less, a vendor is contacted for removal and disposal.

6.0 Equipment and Supplies

6.1 Personal Protective Equipment (PPE)

6.2 Gloves – latex or nitrile

6.3 Safety glasses

6.4 Lab coat

6.5 For handling concentrated acid, the following additional PPE items are used as needed:

6.5.1 Face shield

6.5.2 Acid proof apron

6.5.3 Elbow length acid proof gloves

6.6 Gas chromatograph - Analytical system complete with gas chromatograph suitable for Grob-type injection using capillary columns, and all required accessories including an electron capture detector (ECD), capillary analytical columns, recorder, gases, and syringes. A commercial data system for measuring peak heights and/or peak areas is also used.

6.7 When using an electron capture detector, analyses on two dissimilar columns are necessary to provide confirmation of identifications. The GC is configured for simultaneous dual-column operation using separate injection ports and employing a separate electron capture detector for each column. Columns 1 and 2 are used with the ECD detector.

6.7.1 Column 1 - 30m x 0.32mm ID x 0.50µm, (Rtx®-CLPesticides or equivalent.)

6.7.2 Column 2 - 30m x 0.32mm ID x 0.25µm, (Rtx®-CLPesticides2 or equivalent.)

6.8 Glassware: The glassware used is dependent on the extraction method used. The glassware for a specific method is stated at the beginning of the extraction procedure.

6.9 Amber jars - 1 Liter and 4 oz.

6.10 Repeating pipette - with 5mL and 10 mL tips.

6.11 Pasteur pipette - glass, disposable with bulb.

6.12 Volumetric flask.

6.13 Funnel

6.14 Vials - disposable and 60mL glass.

6.15 Autosampler Vials – 2mL amber glass with open-top screw top cap and Teflon-coated silicon rubber septa.

6.16 Nitrogen Blowdown Device - Turbovap for 60 mL vials capable of temperature control (± 2°C)

6.17 Syringe –gastight.

6.18 pH paper – wide range.

6.19 Analytical Balance

7.0 Standards and Reagents

7.1 Media, solutions, and reagents are prepared, used and stored according to the manufacturer's instructions or the following procedures.

7.2 Reagent Receipt

7.2.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2.2 Documentation for reagents purchased pre-prepared, ready-to-use shall include manufacturer, lot number, type and amount received, date of receipt, opened date, and expiration date.

7.2.3 Documentation for media prepared in the laboratory shall include date of preparation, preparer's initials, type and amount prepared, manufacturer and lot number, and expiration date. All media prepared in the laboratory is documented in the Supplies Preparation Logbook.

7.3 Organic-free reagent water—all references to water in this method refer to organic-free water.

7.4 Concentrated Sulfuric Acid

7.5 Solvents - All solvents should be pesticide quality or equivalent.

7.5.1 Hexane

7.5.2 Dichloromethane

7.5.3 Acetone

7.6 Standards Preparation

7.6.1 All entries and calculation details are documented in the

Standards Preparation Logbook. When available, attach the supplier's "Certificate of Analysis" correctly labeled to facilitate traceability. Print the waterproof label containing:

7.6.1.1 Solution identification number

7.6.1.2 Nominal concentration

7.6.1.3 Expiration date

7.6.1.4 Apply label on the vials and with the help of a permanent marker, place a mark indicating the meniscus.

7.6.1.5 Keep all standard solutions at room temperature inside amber vials.

7.6.2 Good practice: what is removed from the vial never returns to the vial. SGS uses, when available, standards from different manufacturers or a second lot from the same manufacturer, to calculate and verify all calibration standards. If second lots aren't available, SGS prepares a second solution/dilution to use until a second lot can be purchased. SGS has used standard reference material and performance evaluation standards to verify calibration.

7.6.3 Commercial standards come in different solvents; hexane, iso-octane, nonane, just to name a few. SGS practice is to use a solvent compatible with the manufacturer's choice. However, hexane is preferred and will be used whenever possible because of its favorable properties.

7.7 Preparation of Stock Standard Solutions

Stock Calibration Standard Solution Preparation

This standard is prepared the same way for ICALs and CVSs, different lots are used as noted previously. This standard is prepared from commercially available standards as listed in Table 2.

7.8 Preparation of Working Standard Solutions

7.8.1 Working Initial Calibration Standard Solutions Preparation

7.8.1.1 Prepare a minimum of five different concentrations for each parameter of interest, through dilution of the Stock Calibration Standard Solution with hexane (six standards are usually prepared.) Calibration solutions must be replaced after one year, not to exceed the manufactures expiration date, or sooner if comparison with check standards indicates a problem.

7.8.1.2 This standard is prepared from commercially available standards as listed in Table 3.

7.8.2 Working Calibration Verification Standard Solution Preparation

This standard is prepared from the Stock Calibration Standard Solution.

7.8.3 Working Surrogate Solution Preparation

This standard is prepared from commercially available standards as listed in Table 5.

7.8.4 Working Matrix Spike Solution Preparation

This standard is prepared from commercially available standards as listed in Table 6.

7.9 Transfer the standard solutions to vials with PTFE-lined screwcaps. Store at 0-6°C, protected from light. Stock standard solutions are checked frequently for signs of degradation or evaporation, especially immediately prior to preparing calibration standards from them.

7.10 Expiration Dates for Standards

7.10.1 Primary Standard Solutions (ampoule from commercial source): Use the manufacturer's date. If a stability study is in progress, use 5 years.

7.10.2 Stock Standard Solutions (obtained from dilution/mixing Primary Standard Solutions): Up to 5 years, not to exceed the Primary Standard expiration date.

7.10.3 Working Standard Solutions (from Stock/Primary and used during sample processing): Up to 1 year, not to exceed the Stock Standard expiration date.

7.10.4 All solutions prepared by SGS that have a nominal concentration of less than 0.1 ng/µL, are assigned a 30-day expiration time.

7.10.5 Standards will be removed from use and disposed on the expiration date.

8.0 Calibration

8.1 Initial Calibrations

8.1.1 Analyze one solvent blank (hexane) which will pre-condition the GC column and provide a check to determine possible instrument level contamination. The criteria for these blanks are the same as for prep blanks.

8.1.2 Prepare a six-point calibration curve in hexane (a minimum of 5

points) from the Stock Calibration Standard Solution. The PCB and surrogate concentrations are listed in Table 10. Each Aroclor must have its own calibration, on each column. Aroclors 1016 and 1260 may be analyzed together.

8.1.3 One of the standards must be at a concentration near, but above, the method detection limit. Likewise, one of the standards must be at or below the reporting limit. The remaining standards should correspond to the expected range of concentrations found in real samples and define the working range of the GC.

8.1.4 Inject each calibration standard using the same technique as for samples.

8.1.5 The analyst must demonstrate acceptable calibration of the instrument for both columns. This is demonstrated by the evaluation of the relative standard deviation (RSD) of the average response factor (RF) calculated for each individual Aroclor peak which must be less than 20%, (per EPA 8000B) or recalibration of each failing Aroclor is required. Compounds with RSD > 20% require the generation of a new calibration curve. A linear or nonlinear calibration curve (with $r > 0.995$, $r^2 > 0.99$) must be generated when RSD criterion is not met.

8.1.6 If a passing calibration cannot be achieved, then proceed with one or more of the maintenance options as described in Sec. 7.5.11 of EPA 8000B.

8.2 Calibration verification

8.2.1 Analyze the hexane blank which will pre-condition the GC column and provide a check to determine possible contamination. If any peaks appear in the appropriate retention time window for a PCB of interest, check for contamination and if present, reanalyze.

8.2.2 Verify calibration at the beginning of each 12-hour shift by injecting a mid-range calibration verification standard prior to conducting any sample analyses. Standards for Aroclors 1221, 1232, 1242, 1248, 1254, and 1016/1260 are analyzed each day. The measured amount for each analyte in the calibration verification must not exceed ±15% difference when compared to the known amount.

8.2.3 All CCALs in the beginning of the day must be checked for passing. If they do not pass, reanalyze the CCALs or recalibrate as appropriate; otherwise, these Aroclors may not be reported (as “hits” for that day).

8.2.4 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time from the calibration verification standard at the beginning of the analytical shift.

8.2.5 Include a calibration verification standard of Aroclors 1254 and 1016/1260 after each group of 10 samples in the analysis sequence. The measured amount for each analyte in the calibration verification must not exceed ±15% difference when compared to the known amount.

8.2.6 When this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration again and proceeding with sample analysis. (The only exception would be if the samples are non-detect and the instrument is demonstrating increased sensitivity, CVS failing high.) If routine maintenance does not return the instrument performance to meeting the QC requirements based on the last initial calibration, then a new initial calibration for the failing PCBs must be performed.

9.0 Sample Preparation

9.1 Water Sample Preparation

9.1.1 Follow the procedure given in SOP SV_72 (Extraction by SW 3520).

9.2 Solid Sample Preparation

9.2.1 Follow the extraction procedure as directed by SW-846 Test Method 3540C.

9.3 Post-Extraction Sample Preparation— Samples, typically soils and sediments, may need additional preparation. The presence of sulfur and other co-extracting interferences may be removed by following the procedures in SV_138 (Sulfur Cleanup by SW 3660) and SV_363 (Sulfuric Acid Cleanup by Modified Method 3665A).

10.0 Analytical Procedures

10.1 Analytical Procedure

10.1.1 Gas chromatographic conditions

10.1.1.1 Injection volume: 1 µL, splitless

10.1.1.2 Injector temperature: 250°C

10.1.1.3 Detector temperature: 330°C

10.1.1.4 See Table 11 for further details.

10.1.2 Sample Analysis Procedure

10.1.2.1 The following is the required sequencing to be used by the analyst when loading the GC autosampler. Since the analysis is dual column, the analytical sequence is the same for both columns.

10.1.2.2 Verify that the instrument is successfully calibrated. (Passing CVS, etc.)

10.1.2.3 Type the run sequence into the data system.

10.1.2.4 Examine the computer sequence and verify this batch sequence with the sample tray.

10.1.2.5 Start the autosampler.

10.1.2.6 If additional samples are added after the batch sequence has been established, the analyst must verify the run order of the sample tray with the computer sequence. Regardless, the analytical sequence must end with a bracketing CVS.

10.1.2.7 Before emptying the sample wheel, check data runs with run log and sample placement in wheel. This is to ensure that no errors in data acquisition occurred during the sampling sequences.

10.1.3 Qualitative Analysis

10.1.3.1 The identification of PCBs as Aroclors is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes.

10.1.3.2 Qualitative identifications of target analytes are made by examination of the sample chromatograms on dissimilar columns.

10.1.3.3 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed based on a clearly recognizable Aroclor pattern. In order to employ this approach, the analyst must document:

10.1.3.3.1 The peaks that were evaluated when comparing the sample chromatogram and the Aroclor standard.

10.1.3.3.2 The absence of major peaks representing any other Aroclor.

10.1.3.3.3 The source-specific information indicating that Aroclors are anticipated in the sample (e.g., historical data, generator knowledge, etc.).

10.1.3.3.4 Confirmation is necessary when the sample composition is not well characterized.

10.1.4 Quantitative Analysis

10.1.4.1 Calculate the concentration of analyte using each chosen peak from the multi-point calibration.

10.1.4.2 When an analyte concentration is below the PQL, above the MDL, and meets the identification criteria, it is reported with a "J" qualifier as an estimated concentration when requested.

10.1.4.3 When an analyte concentration exceeds the calibration upper limit, it is diluted and reanalyzed. If reanalysis is not possible, the data is reported with an "E" qualifier as an estimated concentration.

11.0 Details of Calculations

11.1 Percent Recovery

Calculate the percent recovery of an extraction/cleanup standard as follows:

$$\%R = \frac{R_{ng}}{S_{ng}} \times 100$$

Where: R_{ng} = amount of extraction standard recovered in ng
S_{ng} = amount of extraction standard spiked in ng

11.2 Percent Relative Standard Deviation (%RSD); To compare three or more replicates, such as Initial Calibration Response Factors, precision expressed as Relative Standard Deviation (RSD) is calculated as follows:

First, calculate the standard deviation (s) of the values to be compared, as follows:

$$s = \sqrt[2]{\frac{\sum_{i=1}^{n}(X - A)^2}{n-1}}$$

Where: n = number of values

Xi; i=1 to n = the individual values
A = average of the values

Then calculate the RSD as follows:

$$RSD = \frac{s}{A} \times 100$$

Where: s= the standard deviation from above
A = average of the values

11.3 Percent (%Drift)

$$\%Drift = \frac{(Calculated[\ ] - Spiked[\ ])}{Spiked[\ ]} \times 100$$

Where: [ ]= is concentration in µg/L or µg/kg

11.4 Target Compound Concentrations

$$Concentration(ug/L) = \frac{(A_s - b)}{m} \times \frac{F_v}{I_v} \times df$$

$$Concentration(ug/Kg) = \frac{(A_s - b)}{m} \times \frac{F_v}{I_v} \times \frac{100}{\%Solids} \times df$$

Where: A_s = Peak area of compound in sample
b = Intercept
m = Slope
F_v = Final volume in liters
I_v = Initial volume in liters
df = Dilution factor

11.5 Correlation Coefficient

The quantity *r*, called the linear correlation coefficient, measures the strength and the direction of a linear relationship between two variables, in this case area and concentration.

$$r = \frac{n\sum xy - \left(\sum x\right)\left(\sum y\right)}{\sqrt{n\left(\sum x^2\right) - \left(\sum x\right)^2}\sqrt{n\left(\sum y^2\right) - \left(\sum y\right)^2}}$$

Where: n = # of ICAL points
x = Concentration
y = area

$COD = r^2$

Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of the determination will equal 1.0. In order to be an acceptable

non-linear calibration, the COD must be greater than or equal to 0.990.

11.6 When determining PCB Aroclors, calculate the response factor (RF) for each Aroclor in the calibration standard using the equation that follows.

$$RF = \frac{A_s}{C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

C_s = Concentration of the analyte or surrogate, in µg/L.

11.7 Surrogate Percent Recovery.

$$\%R = \frac{[found]}{[spiked]} \times 100$$

Where: [*found*] = the concentration measured in the sample
[*spiked*] = the concentration spiked in the sample

11.8 Matrix Spike Recovery.

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

Where: *SSR* = # of ICAL points
SR = Concentration
SA = area

11.9 Relative Percent Difference; to compare the precision of duplicate analysis. Precision expressed as Relative Percent Difference (RPD) is calculated for laboratory sample duplicates, laboratory control sample duplicates, matrix spike duplicates, etc., as follows:

$$RPD = \frac{(C_1 - C_2)}{\left(\frac{(C_1 + C_2)}{2}\right)} x100$$

Where: C_1 = larger of the two values
C_2 = smaller of the two values

12.0 Quality Control Requirements

12.1 All laboratory QC samples are prepared like environmental samples and are taken through the entire preparation and analytical process.

12.2 Practical Quantification Limits (PQL) for this method are listed in Figure 1 and represent the lowest concentration standard used in the initial calibration.

12.3 Quality Control Work Groups (Extraction Batch QC)

SGS defines an extraction batch as no more than 20 samples processed within a 12-hour shift. One laboratory method blank (MB), one Laboratory Control Spike (LCS), one Matrix Spikes and one Matrix Spike Duplicate (MS/MSD) are processed in every extraction batch (workgroup), following the same procedures used for the field samples.

12.3.1 Generally, for MB and LCS samples, soil/solid matrices are replaced by sodium sulfate (Na_2SO_4), water/aqueous matrices by de-ionized water and biological tissues by vegetable oil. An invalid MB or LCS requires a re-extraction of the affected samples.

12.3.2 A Laboratory Control Sample (LCS) must be included with each extraction batch. The LCS consists of an aliquot of a clean (control) matrix like the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

12.3.3 It is the practice of the laboratory to spike the LCS with Aroclor 1254, as it is the most common Aroclor historically detected in the lab. Other Aroclors, such as 1016, 1221, 1242, 1248, 1260, 1262 or 1268, may be used based on request of the client or when specific Aroclors are known to be present or expected in samples.

12.3.4 Client specified samples are used for MS/MSD whenever possible. Whenever sufficient sample for MS/MSD is not available, an LCSD is extracted with the batch.

12.3.5 Documenting the effect of the matrix must include the extraction of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair.

12.4 Sample Quality Control for Analysis (Analytical Batch QC)

12.4.1 An analytical batch is composed of prepared environmental samples which are analyzed together as a group along with their supporting laboratory instrument QC samples. Each analytical batch should include the related extraction batch QC when possible. An analytical batch begins with a passing CVS and

ends with the analysis of a bracketing CVS after 10 samples and within twelve hours.

12.4.2 Before processing any samples, the analyst must demonstrate acceptable calibration of the instrument.

12.4.3 Once the instrument is calibrated, the instrument must be checked for contamination by the analysis of a method blank or solvent blank. The results must show that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a blank must be analyzed as a safeguard against chronic laboratory contamination. The method blanks must be carried through all stages of sample preparation and measurement.

12.4.4 Surrogate recoveries - The laboratory evaluates surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory.

12.5 Additional systematic quality assurance practices

12.5.1 The laboratory participates in performance evaluation studies administered by the QA Office.

12.5.2 Also, when sufficient sample amount is not provided to complete the MS/MSD requirements, an LCSD is added to the batch for monitoring precision.

12.6 Establishing Retention Time Windows

12.6.1 Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window is carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis. The following subsections describe the approach used to establish retention time windows for this method.

12.6.2 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system

conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed.

12.6.3 Record the retention time for each single component analyte and surrogate to three decimal places (e.g., 0.007), in each level of the ICAL. Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate.

12.6.4 If 3x standard deviation of the retention times for a target compound is less than 0.03 (i.e. less than 2 x the sampling rate of the detector), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.03 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).

12.6.5 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the ICAL. If the default standard deviation is employed, the width of the window will be 0.03 minutes.

12.6.6 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

12.6.7 The laboratory must calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument. New retention time windows must be established when a new GC column phase is installed. (The retention time windows should be reported with the analysis results in support of the identifications made.)

12.6.8 If the instrument data system is not capable of employing compound-specific retention time windows, then the analyst may choose the widest window and apply it to all compounds.

12.6.9 The surrogates are added to each sample, blank, and QC sample and are also contained in each calibration standard. Although the surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time

window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

12.7 Method Detection Limit Study and IDOC Schedule

12.7.1 See SOP MI_59 for details and procedures of determining detection limits.

12.7.2 Initial Demonstration of Capability - Each analyst (workcell) must demonstrate initial proficiency with each matrix and sample preparation technique it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. IDOCs are repeated with each relevant change to a technique.

12.8 Control Charts

A system of control charts is applied in this context to track and establish warning and control limits. The objective of these control charts is to reduce errors to required limits. This can help assure that the results have a high probability of being of acceptable, defined analytical quality. For further information on control charts, see SOP MI 140.

12.8.1 Currently limits for LCS recovery are in Table 7.

12.8.2 Control charts are constructed for surrogate standard recovery. Surrogate recoveries for samples and QC samples must be within the control limits developed by the laboratory. Corrective action must be taken if surrogate limits fall outside of control limits. Current surrogate recovery limits are listed in Table 8.

12.8.3 Currently limits for MS/MSD recoveries are set as in Table 9.

12.9 Analytical Documentation Procedures

12.9.1 An instrument directory is created for each batch to organize the associated data. Batch designations are of the format Horizon QUEUE + sequential number. Example: The batch identification number for a PCB batch run on ECD2 on 08/12/21 would be XGC*xxxx*.

12.9.2 Instrument Runlogs

All measurement data is recorded on log sheets in permanent ink. Transcriptions will be avoided whenever possible; the run log is generated by an excel spreadsheet. The record reflects the measurement performed and all appropriate details for conclusions related to the measurement. The record must be initialed and dated by the individual performing the measurement and peer reviewed at the time it is added to the logbook.

12.9.3 Analytical Batch checklist

Analytical batches include LIMS Batch Review Report, all chromatography ran in batch, including instrument QC, batch QC, and samples (in runtime order), and the instrument runlog.

12.9.3.1 ICAL documentation assembled is to include a form 6 type summary form, quant report, and chromatogram for each column for each curve point, and a copy of the runlog.

12.9.3.2 CVS documentation assembled is to include a form 7 type summary form, quant report, and chromatogram for each column, and a copy of the runlog.

12.10 Contingencies for handling out-of-control or unacceptable data

12.10.1 When data are produced that are out-of-control or unacceptable, the client is contacted and informed of the situation. A resolution is agreed upon and carried out. This can involve the request for backup sample, re-sampling to acquire more sample, providing the data qualified and narrated to explain the problems encountered. If a corrective action is needed to solve the problem, it is completed before the samples are re-analyzed.

12.10.2 All data is evaluated by the analysts and data reviewers for compliance with QC objectives. Out of QC control data may be addressed by one or more of the following options:

12.10.2.1 Corrective action - Documentation of corrective actions regarding samples are made in the instrument runlog. These actions may include reanalysis, recalibration, and system maintenance. (A systemic problem or the discovery of a problem affecting multiple instruments necessitates the use of the corrective action form to fully document the investigation and resolution.)

12.10.2.2 Reanalysis of sample

12.10.2.3 Discussion and qualification of data by case narrative

12.10.2.4 Client notification

12.10.2.5 Re-sampling and reanalysis (client decision)

12.10.3 Out of QC control data may be reported if directed by the client if it is qualified by a case narrative detailing the QC problems with advice on the usability of the data.

13.0 Data Review and Reporting Requirements

Please refer to the SOP for Data Review, document number MI_141, for general guidelines. Method specific criteria are listed below.

13.1 ICAL

13.1.1 The ICAL is constructed by average response factor.

13.1.2 A minimum of three peaks for each Aroclor pattern is measured. Up to 5 peaks may be used.

13.1.3 For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor.

13.1.4 The x variable is the concentration of the analyte in the calibration standard aliquot introduced into the instrument. The y variable is the area of the analyte.

13.1.5 The origin (0, 0) is not included as an extra calibration point.

13.1.6 The origin is not forced.

13.1.7 RSD must be less than 20%.

13.1.8 Analysis must not proceed when analytes of interest do not meet ICAL criteria.

13.2 Calibration verification standard (CVS)

13.2.1 Opening CVS

13.2.1.1 A mid-point standard is analyzed daily for each Aroclor to verify calibration prior to sample analysis.

13.2.1.2 The measured concentration of the analytes is compared to the known value. A maximum percent difference of ≤ 15% is allowed for each compound.

13.2.1.3 The CVS must pass SR recoveries in the same manner a sample.

13.2.1.4 Analysis must not proceed when analytes of interest do not meet CVS criteria.

13.2.2 Bracketing CVS

13.2.2.1 A bracketing CVS consisting of Aroclors 1016, 1254, and 1260 must be analyzed at a frequency of every 10 samples.

13.2.2.2 The measured concentration of the analytes is compared to the known value. A maximum percent difference of ≤ 15% is allowed for each compound.

13.2.2.3 The CVS must pass SR as for a sample.

13.2.2.4 Analysis must not proceed when analytes of interest do

not meet CVS criteria.

13.2.2.5 If the measured concentration fails in the CVS and indicates increased sensitivity, samples analyzed prior to the bracketing CVS that do not have hits may be reported with narration.

13.3 Blanks

13.3.1 The method blank is analyzed after the detection system has met required initial calibration verification checks and before sample analysis.

13.3.1.1 The method blank must meet surrogate control limits and at least one of the following criteria.

13.3.1.2 All targets are less than half the PQL.

13.3.1.3 Any compound detected in the blank is less than 10% of the regulatory limit associated with the target.

13.3.1.4 Any compound detected in the blank is less than 10% of the sample result for the same analyte.

13.3.1.5 Corrective action must be taken for any failing blank criteria, see Table 13.

13.4 Laboratory Control Samples (LCS).

13.4.1 The LCS is extracted with each extraction batch and must be spiked with the same standard mix at the same concentration as the MS/MSD.

13.4.2 LCS limits can be found in Table 7. Failure of any of these compounds necessitates corrective action before any samples are reported. If the LCS is outside acceptance criteria, all associated samples must be re-prepped and reanalyzed (See Table 13).

13.4.3 SR recoveries must be within the recovery limits given in Table 8. If the recovery for any SR is not within limits, the following are required: Check all calculations and spiking solutions for accuracy. Reanalyze the LCS if none of the above steps solves the problem. If reanalysis also fails, the related samples must be considered for reanalysis.

13.5 MS/MSD

13.5.1 An MS/MSD must be performed with each extraction batch and analyzed at the same dilution as the parent sample. If there is insufficient sample volume to perform MS/MSD analysis, a LCS duplicate will be analyzed instead.

13.5.2 SR recoveries must be within the recovery limits given in Table

8. If the recovery for any SR is not within limits, the following are required: Check all calculations and spiking solutions for accuracy. Look for a confirming trend in the sample analysis. Reanalyze the MS/MSD if none of the above steps solves the problem, report the data with narration.

13.5.3 The limits for matrix spike compound recovery and relative percent difference (RPD) are given in Table 9. If the criteria fail, proceed as follows:

13.5.3.1 Evaluate the sample spiked. Highly contaminated or non-homogenous matrices may not be amenable to achieving the spiking limit requirements. See Table 13 for corrective action.

13.5.3.2 Information from the MS/MSD is sample or matrix specific and is not used for batch validity determination. Report the failures to the client whose sample was spiked. Qualify results as necessary for that sample only.

13.5.4 A single spike with an un-spiked duplicate can be analyzed in place of the MS/MSD pair if adequate sample volume is not available. The addition of an LCSD is recommended.

13.6 Sample QC

13.6.1 Surrogate Standards (SR)

13.6.1.1 SR recoveries must be within the recovery limits given in Table 8. If the recovery for any SR is not within limits, the following are required:

13.6.1.2 If one surrogate passes criterion, report data with narration. Specific to GE Pittsfield, MA, the failing surrogate must still be within the ranges of 10%-200% or re-extraction is required. If both surrogates fail outside the acceptance window, proceed to the next step.

13.6.1.3 Check all calculations for accuracy, spiking solutions, and internal standards.

13.6.1.4 Check for matrix interference. Re-extraction is required to confirm if observed.

13.6.1.5 Re-extract and re-analyze the sample if none of the above steps solves the problem.

13.6.1.6 If re-extraction and re-analysis also fails, matrix interference may be assumed as the cause. The original

run is reported with flags and the confirmation of surrogate failure is narrated.

13.7 Laboratory Sample Duplicates: One un-spiked duplicate is analyzed upon client request.

14.0 Preventative Maintenance

14.1 Instrument Maintenance

14.1.1 All instrumentation is serviced by an external instrumentation service vendor or by SGS personnel trained in preventative maintenance. Preventative maintenance is performed at scheduled intervals on all equipment. All instrument preventative maintenance is performed according to the manufacturers recommended procedures. All maintenance shall be thoroughly documented in the instrument maintenance log. Suggestions of frequency are only a guide. For example, if the need arises to make manual injections, the GC injection port septa may need changing more often.

14.1.2 Gas Chromatograph

14.1.2.1 Corrective: Septa change and check tank pressure.

14.1.2.2 Weekly: Check the tightness of liner and column nuts when using temperature-programmed analysis. Cool the inlet and column oven and be sure to reduce the inlet pressure. There should be 1mm between the septum nut and the c-ring once retightened. Change the liner, o-ring, split seal, washer, clip the column and reinstall with a new ferrule as needed.

14.1.2.3 Monthly: Inspect, clean and/or replace the split line trap. Renew gas cylinders. With typical usage, A-size cylinders will supply for about 2 weeks. Replace sooner if pressure falls below 500psi.

14.1.2.4 Annually: Recondition or replace internal and external traps and chemical filters.

14.1.3 Electron Capture Detector

14.1.3.1 Semi-annually: Wipe test for radiation leakage.

14.1.4 Autosampler

14.1.4.1 Daily: Check the level solvent in the rinse vials and dispose of the waste in the waste vial.

14.1.4.2 Semiannually: Wipe off old lubricant and dust. Then

re-lubricate moving parts.

14.1.5 Computer and Printer

14.1.5.1 Weekly: Check disk space.

14.1.5.2 Semiannually: Clean printer. Check output assembly on printer.

14.2 Equipment Taken Out of Service

14.2.1 When equipment is removed from service for repair, the equipment shall be plainly marked as having been taken out of service. Use the signs provided. This equipment will not be used until the repair activities are complete and the instrument meets any applicable performance specifications.

14.3 Corrective maintenance

14.3.1 Corrective maintenance is deemed necessary when the analytical system cannot meet calibration criteria or other specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, column replacement, or detector component replacement. All corrective maintenance is performed according to the manufacturers recommended procedures by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log.

15.0 Tables

15.1 Table 1—Detection Limits

CAS. No.	Aroclor	µg/L
12674-11-2	1016	1
11104-28-2	1221	1
11141-16-5	1232	1
53469-21-9	1242	1
12672-29-6	1248	1
11097-69-1	1254	1
11096-82-5	1260	1

Chemical Abstract Service Registry Number

15.2 Table 2—ICAL and CCAL Stock prep guide

Solution Name	Initial Concentration µg/mL	Initial Volume mL	Final Volume mL	Final Concentration µg/mL
Aroclor	1000	0.080	20	4.0
TCMX/DCBP	200	0.040	20	0.400

Prepared in Hexane

15.3 Table 3—Initial Calibration (working levels) prep guide

Solution Name	Initial Concentration µg/L	Initial Volume mL	Final Volume mL	Final Concentration µg/L
ICAL 2000	4000	0.5	1	2000
ICAL 1000	4000	0.25	1	1000
ICAL 500	4000	0.125	1	500
ICAL 200	4000	0.05	1	200
ICAL 100	4000	0.025	1	100
ICAL 40	4000	0.01	1	40

Prepared in Hexane

15.4 Table 4—Calibration Verification (working levels) prep guide

Solution Name	Initial Concentration µg/mL	Initial Volume mL	Final Volume mL	Final Concentration µg/mL
Aroclor	4000	0.250	1.0	1000
TCMX	400			100
DCBP	400			100

Prepared in Hexane

15.5 Table 5—Surrogate spike prep guide

Solution Name	Initial Concentration µg/mL	Initial Volume mL	Final Volume mL	Final Concentration µg/mL
TCMX/ DCBP	200	1	200	1

Prepared in acetone

15.6 Table 6—Matrix spike prep guide

Solution Name	Initial Concentration µg/mL	Initial Volume mL	Final Volume mL	Final Concentration µg/mL
Aroclor	1000	1	100	10

Prepared in acetone

15.7 Table 7—LCS recovery limits

LCS Analyte	Water (%)
Aroclor 1254	70-130

With RPD limits of 24.3%

15.8 Table 8—Surrogate standard recovery limits

Surrogate Compound	8082 Water (%)	608 Water (%)	Soil (%)
TCMX	40-120	60-140	40-120
DCBP	40-120	60-140	40-120

15.9 Table 9—MS/MSD spike recovery limits for method 8082A

MS/MSD Compound	Water (%)	Soil (%)
All Aroclors	30.9-150	40.8-116

With RPD limits of 25.6%

15.10 Table 10

AROCLOR	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)
1016	40	100	200	500	1000	2000
1221	40	100	200	500	1000	2000
1232	40	100	200	500	1000	2000
1242	40	100	200	500	1000	2000
1248	40	100	200	500	1000	2000
1254	40	100	200	500	1000	2000
1260	40	100	200	500	1000	2000
Surrogates						
TCMX	4	10	20	50	100	200
DCBP	4	10	20	50	100	200

15.11 Table 11—GC Conditions

Start Temp (^{0}C)	End Temp (^{0}C)	Rate (^{0}C/min)	Time (Min)
120	120	0	0.50
120	200	45	1.78
200	230	14	2.00
230	330	30	3.33
230	330	0	2.00

	Column	Carrier Flow
	RTX-CLPest	4.9 mL/min
	RTX-CLPest2	2.9 mL/min

15.12 Table 12 – Method 608 Acceptance Criteria

Aroclor	Calibration Verification	Range for DOCs (%)	Range for LCS (%)	Maximum MS/MSD RPD %
PCB-1016	75 - 125	61 - 103	50 - 140	36
PCB-1221	75 - 125	44 - 150	15 - 178	48
PCB-1232	75 - 125	28 - 197	10 - 215	25
PCB-1242	75 - 125	50 - 139	39 - 150	29
PCB-1248	75 - 125	58 - 140	38 - 158	35
PCB-1254	75 - 125	44 - 130	29 - 140	45
PCB-1260	75 - 125	37 - 130	8 - 140	38

15.13 Table 13—Method 8082 QC Chart

QC Type	Frequency	Criteria	Deficiency	Flag	Flag applied to	Corrective Action
ICAL	As needed to maintain acceptable CVS, or after major maintenance	RSD < 20%	RSD > 20%	(J)	Failing compounds	Do not begin sample analysis without acceptable calibration for each compound of concern
CVS	Daily from a second source before sample analysis	Maximum %D of 15%	%D > 15%	(R)	All samples	Do not begin sample analysis without acceptable calibration for each compound of concern
	Bracket for each 10 samples analyzed	Maximum %D of 15%	%D > 15% (high sensitivity)	(J)	Failing compounds	Report data with narration
			%D > 15% (low sensitivity)	(J)	Failing compounds	Re-analyze samples
Method Blank	One per 24h/batch/amtrix	Each compound < half SOP PQL or < 10% of level in sample	Any compound failing criteria and is detected in the sample	–	–	Re-extract all samples (Re-analyze all samples if instrument is suspected)
			# of MEs are allowable, compounds are NOT detected in the sample above the RL (MDL or RL)	–	–	Report data with narration
			# of MEs > allowable and these compounds are NOT detected in the sample above the RL (MDL or RL)	–	–	Re-extract all affected samples
Instrument Blank	After every CVS set.	Same as method blank	Instrument blank not analyzed	See method blank	See method blank	A sample that meets the blank criteria for the compound in question will suffice. Otherwise, re-analyze all affected samples.
LCS	One per extraction batch	Recovery within control limits	%R > UCL, # MEs are allowable, and compounds are NOT detected in the sample above the RL (MDL or RL)	(UJ+)	–	Report data with narration
			%R > UCL, # MEs > allowable, and compounds are NOT detected in the sample above the RL (MDL or RL)	–	–	Re-extract and re-analyze samples
			%R > UCL, # MEs are allowable, and compounds ARE detected in the sample above the RL (MDL or RL)	(J+) for positive results	Effected compounds only	Report data with narration
			%R > UCL, # MEs > allowable, and compounds ARE detected in the sample above the RL (MDL or RL)	–	–	Re-extract and re-analyze samples
			%R < LCL, # MEs are allowable, and compounds are NOT detected in the sample above the RL (MDL or RL)	–	–	Re-extract and re-analyze samples, contact client for more sample
			%R < LCL, # MEs > allowable, and compounds are NOT detected in the sample above the RL (MDL or RL)	–	–	Re-extract and re-analyze samples, contact client for more sample
			10% < %R < LCL, # MEs are allowable, and compounds ARE detected in the sample above the RL (MDL or RL)	(J-) for positive results	Effected compounds only	Report data with narration
			%R < LCL, # MEs > allowable, and compounds ARE detected in the sample above the RL (MDL or RL)	–	–	Re-extract and re-analyze samples, contact client for more sample
			10% < %R < LCL, # MEs are allowable, and compounds are NOT detected in the sample above the RL (MDL or RL) AND LCSD %R is acceptable	(J)	Effected compounds only	Report data with narration

QC Type	Frequency	Criteria	Deficiency	Flag	Flag applied to	Corrective Action
LCSD	One per extraction batch when MS/MSD has limited volume	RPD within control limits	RPD > CL	#	Failing compounds	Flag compounds for evaluation of MS/MSD RPDs and compound precision. Report data with narration
MS/MSD	One pair per extraction batch	%R within CLs and RPD within CL	MS or MSD %R > UCL Or MS or MSD %R < UCL Or MS/MSD RPD > CL	#	Failing compounds in MS/MSD report	Report data with narration
Duplicate sample (DUP)	One per extraction batch when batch has 10 or more samples	RPD < 35% when both detects are ≤ 5X the RL and RPD < 20% when both detects are ≥ 5X the RL	RPD exceedance	#	Failing compounds in DUP report	Flag failures, report data with narration
Surrogate (SR)	Every sample, including QC samples	Recovery within control limits	SR failure within 10% of limits	(J)	–	Re-analyze sample. Report re-analysis if passing criteria are achieved. Otherwise, re-extract sample
			High recovery failure, more than one SR out.	(J+) or (UJ+)	–	Re-extract sample
			Low recovery failure	(J-)	–	Re-extract sample. Report re-extraction if passing criteria is achieved. Report original run if a confirming trend is observed, include raw data for both.
Equipment Blank	Client controlled, usually 1/20 of field samples	Same as method blanks	Any	(J+) for positive results	–	Report data with narration
Holding time	All samples	7-14 days, see SOP for specific requirements	Any	(R)	All analytes in the sample	Notify client. If requested to do so, report data with narration.
Preservation	All samples	0-6°C	Any	(J) positive results (R) or (UJ) for non-detects	All analytes in the sample	Notify client. If requested to do so, report data with narration.

The number of allowable ME is determined as follows (approx. 5%):

# of permitted ME =	> 110 analytes in LCS, no more than 6
	91-109 analytes in LCS, no more than 5
	71-90 analytes in LCS, no more than 4
	51-70 analytes in LCS, no more than 3
	31-50 analytes in LCS, no more than 2
	11-30 analytes in LCS, no more than 1
	< 11 analytes in LCS, none allowed

Acronyms

CL =	Control Limit
UCL =	Upper Control Limit
LCL =	Lower Control Limit
RL =	Reporting Limit
ME =	Marginal Exceedance
MDL =	Method Detection Limit

16.0 Definitions

16.1 RPD – Relative Percent Difference

16.2 RSD – Relative Standard Deviation

16.3 MB – Method Blank

16.4 LCS – Laboratory Control Spike

16.5 LCSD – Laboratory Control Spike Duplicate

16.6 MS – Matrix Spike

16.7 MSD – Matrix Spike Duplicate

16.8 DUP – Duplicate

16.9 MDL – Method Detection Limit

16.10 PQL – Practical Quantitation Limit

16.11 CVS – Calibration Verification Standard

16.12 ECD – Electron Capture Detector

16.13 PPE – Personal Protective Equipment

16.14 GC – Gas Chromatography

16.15 MSDS – Material Safety Data Sheet

16.16 RF – Response Factor

16.17 SS – Surrogate Standards

16.18 IDOC – Initial Demonstration of Capability

16.19 See SGS document DC_139 Appendix D for a full list of definitions

17.0 References

17.1 SW-846 Method 8082A “Polychlorinated Biphenyls (PCBs) by Gas Chromatography” Revision 1 February 2007

17.2 SW-846 Method 3520C Revision 3, December 1996

17.3 SW-846 Method 3540 Revision 0, December 1996

17.4 SW-846 Method 8000B Revision 2, December 1996

17.5 SGS document DC_139 Appendix D Definitions

17.6 SGS document MI_254 Chemical Hygiene Plan

17.7 SGS document MI_59 Determination of Method Detection Limits

17.8 SGS document SV_72 Method 3520

17.9 SGS document SV_138 3660 Sulfur Cleanup

17.10 SGS document SV_363 Sulfuric Acid Cleanup (Modified Method 3665A)

17.11 SGS document MI_2 Glass Prep

17.12 EPA 608.3 “Organochlorine Pesticides and PCBs by GC/HSD” December 2014

Attachment 4

AECOM Electronic Data Deliverable Specification

AECOM Electronic Data Deliverable Specification

Documentation of the structure and contents of the EDD is now provided directly by the EQuIS Data Processor (EDP). Click the **EDD Description** button in the **Tools** section of the **Home** ribbon section of EDP. The AECOM format file and EDP software (for data providers that do not have it already) are available from http://www.earthsoft.com/products/edp/edp-format-for-aecom/ The format will have to be "registered" when first launched in EDP.

Each EDD will comprise 4 files, to describe samples, tests, results, and batches. The format file has two different sections for samples, Field and Lab, only one of which can be included in the EDD. Which sample section to use will be communicated by the AECOM data manager at project setup.

Submittal

The EDD file can be in one of the following formats:

- ZIP archive of tab-delimited text files (.txt)
- spreadsheet (.xls or .xlsx)
- database (.mdb)

Regardless of the method of EDD Submittal, EDD Packages must be named using a specific naming convention.

EDD File Name:

<Unique ID>.<Facility Code>.AECOM.{zip | xls | xlsx | mdb}

ZIP archive text file EDD section names:

<Unique ID>.<EDD Section Name>.txt

Or XLS worksheet *or* MDB table EDD section names:

<EDD Section Name>

Where:

<Unique ID> = A unique identifier which will be the Sample Delivery Group name unless other arrangements have been made.

<Facility Code> = The facility code for the facility to which this EDD will be loaded, will be communicated by the AECOM data manager at project setup.

<EDD Section Name> = The name of the section within the EDD (i.e. AECOMLabSMP or AECOMFSample, AECOMLabTST, AECOMLabRES, AECOMLabBCH) as it appears in EDP.

Between each of the name elements is a "." (period). It is very important that it is a period and not a "-" (dash), "_" (underscore), or any other character.

Resubmittal

EDD packages may be resubmitted. However, in order to resubmit corrected EDDs, the files must each be renamed, regardless of the reason(s) for resubmittal.

Example: A lab originally submits an EDD Package (.zip) file named "20100129.MySite.AECOM.zip" which contains EDDs named "20100129.AECOMFSample.txt," etc. If the lab later makes a change to one of the EDDs, it would have to submit a new EDD Package named "20100129R.MySite.AECOM.zip" with EDDs named "20100129R.AECOMFSample.txt," etc.

Reference Values

A Reference Values file should be delivered from the AECOM data manager to the data provider at project setup. No EDDs will be accepted that do not strictly adhere to the project-specific reference values. If new values need to be used, they must be identified and explained to the AECOM data manager who will provide approval or alternate codes to use before any EDD should be submitted.

Attachment B

Example of Field Data Sheet for the Collection of Biological Monitoring Data

18.1 FIELDSHEET FOR THE COLLECTION OF BIOLOGICAL MONITORING DATA

New York State Department of Environmental Conservation

FIELD DATA SHEET 4-letter identifier _____

STREAM / STATION _____ **CITY/TOWN/VILLAGE** _____
ROUTE NO. _____
DATE _____
TIME : ARRIVAL _____ **UNIQUE FEATURES** _____
DEPARTURE _____
COLLECTORS _____ **SITE TYPE:** **RIBS SCREENING** _____
LATITUDE\ LONGITUDE _____ **RIBS INTENSIVE** _____
MULTI-SITE SURVEY _____

PHYSICAL AND CHEMICAL PARAMETERS

DEPTH (meters) _____ TEMPERATURE (°C) _____
WIDTH (meters) _____ SPEC. CONDUCT. (µmhos) _____
CURRENT (cm/sec) _____ pH _____
CANOPY (%) 0 10 25 50 75 90 100 D.O. (mg/l; ppm) _____ / sat. _____ %
EMBEDDEDNESS (%) _____ SALINITY _____
SECCHI DISK _____

SUBSTRATE: (%) Rock _____ Rubble _____ Gravel _____ Sand _____ Silt _____

AQUATIC VEGETATION: Algae (suspended) _____ Algae (filamentous) _____

Diatoms (on rocks) (%) _____ Thickness _____ Macrophytes (%) _____

TYPE OF SAMPLE		OCCURRENCE OF MACROINVERTEBRATES			
Multiplate	_____	Ephemeroptera	_____	Chironomidae	_____
Kick, sample retained	_____	Plecoptera	_____	Simuliidae	_____
Kick, sample not retained	_____	Trichoptera	_____	Decapoda	_____
Ponar	_____	Coleoptera	_____	Gammaridae	_____
Organisms for toxics	_____	Megaloptera	_____	Mollusca	_____
Photograph	_____	Odonata	_____	Oligochaeta	_____
Microtox sample	_____	Other	_____	_____	
Other	_____				

FAUNAL CONDITION: very good _____ good _____ poor _____ very poor _____
Habitat: adequate _____ impoundment _____ headwater _____ sand _____ gravel _____
bedrock _____ low flow _____ other _____
Landuse: Residential _____ Agriculture _____ Commmerical _____ Industrial _____
Forest _____ Recreational _____ Wetland _____

NOTES, OBSERVATIONS	RIBS SCREENING SITE CRITERIA
	1. Mayflies (3 or more taxa) _____
	2. Stoneflies (present) _____
	3. Caddisflies (less abund. than mayflies) _____
	4. Beetles (present) _____
	5. Worms (sparse or absent) _____