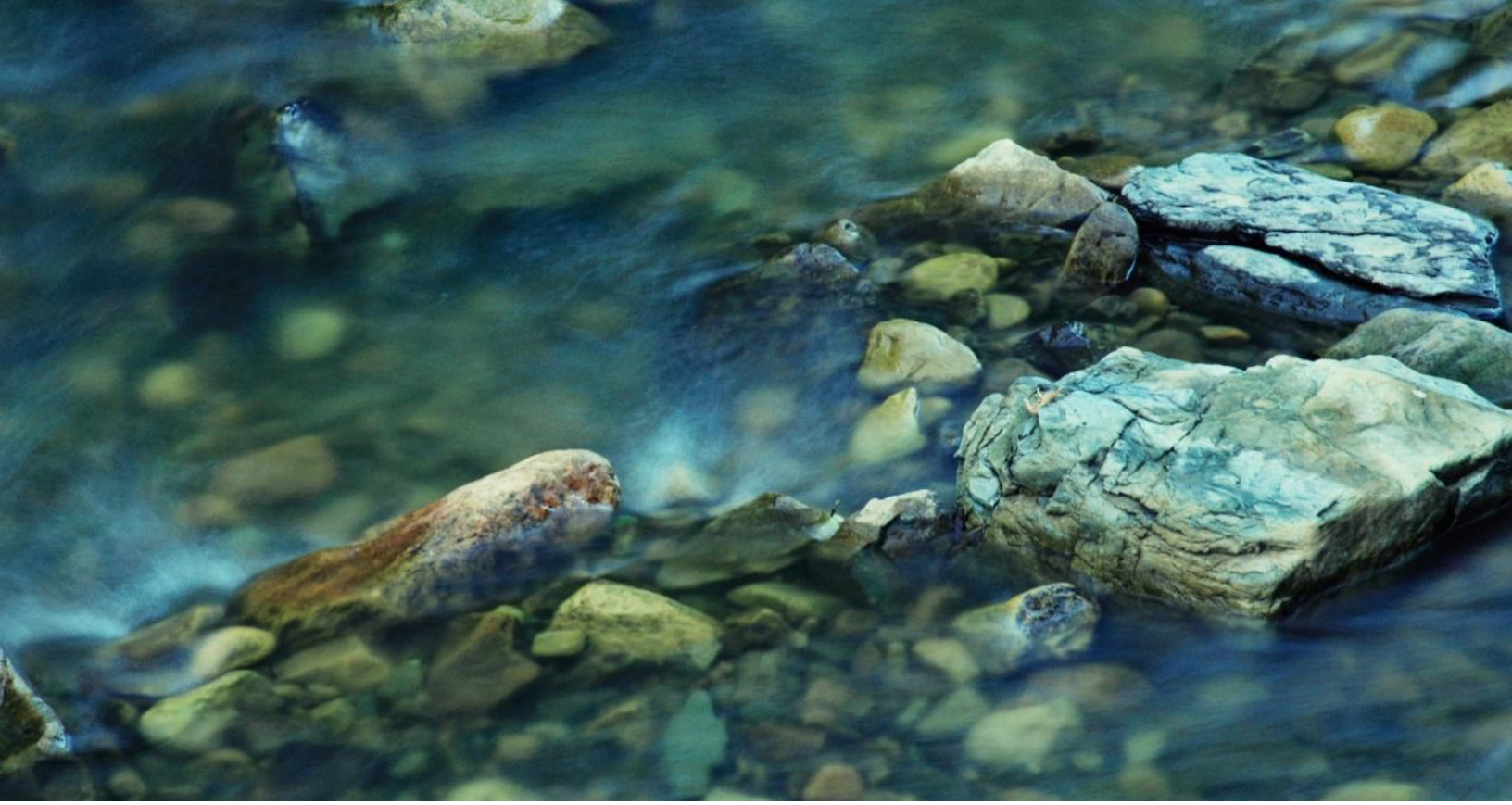

Revised July 2021
Durez Inlet

ANCHOR QEA

# Aquatic Assessment Work Plan

Durez Inlet
NYSDEC Site #932018
North Tonawanda, New York

Prepared for Glenn Springs Holdings, Inc.

Revised July 2021
Durez Inlet

# Aquatic Assessment Work Plan

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# ABBREVIATIONS

| | |
|---|---|
| $^{13}C$ | carbon-13 |
| CCV | continuing calibration verification |
| City | City of North Tonawanda |
| cm | centimeter |
| COC | chain-of-custody |
| Cs-137 | Cesium-137 |
| D/F | Polychlorinated dibenzo-*p*-dioxins and furans |
| DEC | New York State Department of Environmental Conservation |
| DQO | data quality objective |
| Durez NT | Durez North Tonawanda |
| EDD | electronic data deliverable |
| EPA | U.S. Environmental Protection Agency |
| GSH | Glenn Springs Holdings, Inc. |
| $K_{oc}$ | organic carbon partitioning coefficient |
| LCS | laboratory control sample |
| LDPE | low-density polyethylene |
| MECP | Ontario Ministry of Environment, Conservation and Parks |
| MSD | matrix spike duplicate |
| ng/kg | nanograms per kilogram |
| OPR | ongoing precision and recovery sample |
| OCC | Occidental Chemical Corporation |
| Pb-210 | Lead-210 |
| PCF | Pettit Cove Flume |
| PRC | performance reference compound |
| QA | quality assurance |
| QC | quality control |
| SGV | Sediment Guidance Value |
| SOP | standard operating procedure |
| SRM | standard reference material |
| TCDD | 2,3,7,8-tetrachlorodibenzodioxin |
| TEF | toxic equivalency factors |
| TEQ | toxic equivalency |
| TOC | total organic carbon |
| USACE | U.S. Army Corps of Engineers |
| WHO | World Health Organization |

# 1 Introduction

The Durez North Tonawanda (Durez NT) manufacturing facility operated from 1926 until its closure in 1995. Until 1993, non-contact cooling water and stormwater from the Durez NT facility was discharged into the Pettit Cove Flume (PCF), a City of North Tonawanda (City) storm sewer draining the central portion of the City. The PCF discharges into Pettit Cove, a half-acre triangular-shaped, manmade embayment of the Little Niagara River (a tributary of the East Branch of the Niagara River; Figure 1-1). Polychlorinated dibenzo-*p*-dioxins and furans (D/F) potentially associated with the historic discharges from the Durez NT facility have been detected in sediments within the PCF, as well as in Pettit Cove and the Niagara/Little Niagara River.

Between 1977 and 2014, Occidental Chemical Corporation (OCC) and Glenn Springs Holdings, Inc. (GSH) implemented extensive source control actions at and around the Durez NT facility and removed accumulated sediments from both the PCF and Pettit Cove. Section 1.1 of this Work Plan provides a summary of prior source control and remediation activities. Section 1.2 provides a summary of historical D/F sediment characterization data collected within Pettit Cove and the Niagara/Little Niagara River.

Following completion of source control actions at the Durez NT facility, the New York State Department of Environmental Conservation (DEC) requested that GSH characterize the nature and extent of sediment D/F concentrations and associated environmental risks in Pettit Cove and the Niagara/Little Niagara River. GSH subsequently submitted a draft *Work Plan for Aquatic Assessment and Additional Sediment Monitoring* (Work Plan; GHD 2020). Addressing DEC comments on the earlier draft Work Plan and consistent with discussions between DEC and GSH through June 2021, the final Work Plan is detailed below.

D/F concentrations in recent surface sediment samples collected from Pettit Cove and the Niagara/Little Niagara River (including at upstream background locations) exceed Sediment Guidance Values (SGVs) specified in DEC guidance for *Screening and Assessment of Contaminated Sediment* (DEC 2014).[1] When exceedances of the SGVs are noted, that same guidance recommends collection of site-specific data to reduce uncertainty and develop a site-specific SGV.

Specifically, SGVs for D/F were developed in the DEC guidance using an equilibrium-partitioning (EqP) based approach. This approach utilized standard default assumptions for organic carbon partitioning coefficients ($K_{OC}$'s). However, as Section 9 of the DEC (2014) guidance suggests, $K_{OC}$ can

[1] Table 5 of DEC (2014) cites a preliminary freshwater sediment guidance value (SGV) of 0.5 nanograms per kilogram (ng/kg; dry weight basis) for 2,3,7,8-tetrachlorodibenzodioxin (TCDD) and other D/F congener toxicity equivalents. Sediment D/F concentrations below this value are considered to present little/no risk (Class A); sediment D/F concentrations exceeding this value (Class B or C) require further assessment of potential toxicity.

vary substantially depending on site-specific conditions. The DEC guidance describes a process for incorporating site-specific information to develop site-specific EqP-based SGVs.

Consistent with the DEC (2014) guidance, this Work Plan describes an approach to the aquatic assessment that includes the following: 1) data collection to develop a site-specific D/F SGV; 2) surveys of the current benthic and fish communities within Pettit Cove and collection of sediment and tissue samples to support additional characterization of D/F concentrations in these media; and 3) data collection to verify source control effectiveness and characterize natural recovery processes and rates. If the data collected pursuant to this Work Plan suggest that additional data are needed to complete characterization of the nature and extent of sediment D/F concentrations and/or associated environmental risks in Pettit Cove and the Niagara/Little Niagara River, then the scope of any additional sampling will be provided in a future addendum to this Work Plan as necessary.

## 1.1 Previous Source Control and Remediation Activities

As discussed in Section 1, between 1977 and 2014 OCC and GSH implemented extensive source control and remediation actions at the Durez NT facility. These actions included characterization and remediation of potential D/F sources to the PCF and Pettit Cove; cleaning, lining and rerouting sewer lines that previously discharged from the Durez NT facility; and sediment removal from Pettit Cove. A summary of source control and remediation activities is as follows:

- 1977: An engineered clay cap was constructed over a portion of the Durez NT facility.
- 1989: A granular activated carbon treatment system was installed to treat stormwater prior to discharge to the PCF.
- 1990: An interceptor trench was constructed around the perimeter of the Durez NT facility.
- 1992: Durez NT facility and downstream City storm and sanitary sewers were cleaned.
- 1993: Portions of the PCF were re-routed by the City to the Walck Road sewer system, and the Walck Road outfall relocated to its current location in the Niagara River.
- 1995: Sediments were dredged from Pettit Cove and a clay liner/rip-rap cap constructed on the post-dredge surface; a cutoff wall was also installed to contain dense non-aqueous phase liquids in the subsurface of adjacent Pettit Cove property.
- 2000: Approximately 300 tons of sediment that had accumulated above the clay liner/rip-rap cap constructed in 1995 were dredged; tar was also removed from the PCF.
- 2010: Several bulkheads located in the Walck Road and Wilson Avenue sewer systems were repaired, further controlling drainage from the Durez NT facility to the storm sewer.
- 2012: Sediments were removed from the eastern drainage ditch at the former Durez NT plant and areas of the Durez NT plant were regraded to prevent surface runoff from migrating off site.

- 2014: Approximately 215 tons of sediment and debris were removed from the PCF, Walck Road, Wilson, and Harding Avenue sewers; the Nash Road portion of the PCF was subsequently lined along with portions of three laterals.
- 2016: The Walck Road storm sewer adjacent to the Durez NT facility was lined, including manholes, and several bulkheads were repaired.

Pettit Cove source controls from the former Durez NT facility were completed in 2014.

## 1.2 Aquatic Sampling Data Collected Post-Source Control

This section provides a summary of aquatic sediment and biota data collected in Pettit Cove and the Niagara/Little Niagara River after completion of source control efforts in 2014. GSH collected surface (0 to 10 centimeters [cm]) sediment samples at 11 locations within the Little Niagara River and in Pettit Cove, including: 1) three locations in the Little Niagara River upstream of Pettit Cove in 2016; 2) three locations in Pettit Cove that were subject to quarterly monitoring between August 2017 and May 2019; and 3) five locations in the Little Niagara River downstream of Pettit Cove in 2017 (three located at approximately mid-channel and two located along the eastern shoreline; Figure 1-2).[2]

The Ontario Ministry of Environment, Conservation and Parks (MECP) also collected surface (0 to 3 cm) sediments and deployed caged mussels at one location in Pettit Cove[3], two locations in the Little Niagara River (one upstream and one downstream of Pettit Cove), and two locations in the coves at Fisherman's Park (downstream Niagara River) in 2015 and 2018 (Figure 1-2; Ontario Ministry of Environment 2018).[4]

An evaluation of these recent data is presented in Section 2.

## 1.3 Objectives of Aquatic Assessment

The primary objectives of the aquatic assessment described in this Work Plan are as follows:

- Verify that upland source controls implemented at the Durez NT site over the last 40 years have controlled D/F inputs to Pettit Cove and the Niagara/Little Niagara River.
- Characterize the bioavailability of D/F in aquatic sediments and representative biota tissues in Pettit Cove and Fisherman's Park and determine if site-related contaminants have impacted fish and wildlife resources.
- Characterize natural recovery rates and processes (e.g., net sedimentation rates and bioturbation depths) in Pettit Cove and Fisherman's Park.

[2] Note that three locations were originally targeted along the eastern shoreline (NS-1, NS-2, and NS-3 shown in Figure 2-1); however, no sample was collected at location NS-3 because no sediment was present at this location.
[3] MECP added a second location in Pettit Cove in the 2018 sampling event.
[4] MECP has been sampling surface sediments and caged mussels in the vicinity of Pettit Cove approximately every three years since 1991. The locations at Fisherman's Park were added in 2012.

Specific data quality objectives (DQOs) are provided in Section 3.1.

## 1.4 Organization of Work Plan

The remainder of this work plan is organized as follows:

- Section 2 provides a summary and evaluation of the existing aquatic sediment and biota D/F data, including a preliminary conceptual understanding of the origin, fate, transport, and bioaccumulation of D/F within Pettit Cove and the Niagara/Little Niagara River.
- Section 3 describes the proposed sampling and analysis plan for this project which includes: 1) DQOs; 2) the proposed sampling and analysis scope of work; 3) a summary of field sample handling, packaging, and shipping requirements; 4) a description of the analytical methods to be used; 5) a summary of field and laboratory quality assurance (QA) and quality control (QC) requirements; 6) a summary of documentation, recordkeeping, and reporting requirements; and 7) a description of health and safety requirements.
- Section 4 provides a proposed schedule for completion of the scope of work described herein, and a description and schedule for the final report summarizing the results of the aquatic assessment.

# 2 Preliminary Conceptual Site Model

## 2.1 Evaluation of Existing Aquatic Dioxin/Furan Data

Section 1.2 provided a summary of aquatic sediment and caged mussel D/F data collected in Pettit Cove and the Niagara/Little Niagara River since 2014 (i.e., post-source control). The evaluations presented in this section focus primarily on these more recent data sets, as they pertain to the development of a contemporary understanding of D/F sources and fate and transport. However, additional (pre-2014) historical sediment and caged mussel data have been included to evaluate changes in D/F concentrations over time. It should be noted that the analyses presented in this section utilize toxic equivalency (TEQ) concentrations calculated for D/F congeners using the 2005 World Health Organization (WHO) mammalian toxic equivalency factors (TEFs; Van den Berg et al. 2006).

### *2.1.1 Sediments*

Figure 2-1 summarizes D/F total TEQ[5] concentrations in surface sediments (0 to 10 cm) at the 11 locations sampled by GSH within the Little Niagara River and in Pettit Cove since 2014 (sampling locations are shown in Figure 1-2), revealing the following:

- Recent D/F concentrations in Little Niagara River surface sediments located upstream of Pettit Cove range from approximately 5 to 10 nanograms per kilogram (ng/kg; dry weight basis) TEQ, exceeding the preliminary SGV of 0.5 ng/kg TEQ. These sampling locations represent regional background levels removed from potential discharges from the Durez NT facility.
- Recent surface sediment D/F concentrations within Pettit Cove are higher than regional background levels and generally range from approximately 500 to 50,000 ng/kg TEQ. While there appears to be a concentration decline over the 2-year quarterly monitoring period, the variability in these data confound the downward trend.
- Recent surface sediment D/F concentrations in the Little Niagara River nearshore area immediately downstream of Pettit Cove (approximately 2,000 to 5,000 ng/kg TEQ) are similar to those in Pettit Cove. Surface sediment D/F concentrations decline significantly at the downstream mid-channel locations (approximately 10 to 50 ng/kg TEQ) and are only slightly elevated above regional background levels.
- For reference, Figure 2-1 also includes horizontal lines showing recent surface sediment D/F concentrations measured by MECP in Pettit Cove and the Niagara/Little Niagara River. The depth of sediment collected by MECP (0 to 3 cm) is shallower than the samples collected by GSH (0 to 10 cm). Nevertheless, surface sediment D/F concentrations have generally been consistent between these two monitoring programs.

[5] Sediment TEQ concentrations based on WHO 2005 mammal toxicity equivalency factors (TEFs).

- MECP sampling conducted in the two coves at Fisherman's Park reveal recent surface sediment D/F concentrations lower than Pettit Cove, ranging from approximately 300 to 800 ng/kg TEQ.

The time series of surface sediment D/F concentrations in Pettit Cove, including all data collected by DEC, MECP, and GSH since 1991, is presented in Figure 2-2.[6] Surface sediment D/F concentrations in Pettit Cove appear to have declined roughly 100-fold over the last 30 years, likely in response to upland source control and remediation activities (Section 1.1).

The average surface sediment D/F congener TEQ profiles (or fingerprints) upstream and downstream of Pettit Cove are presented in Figure 2-3. Congener TEQ profiles are similar between Pettit Cove (Panel b), nearshore sediments immediately downstream of Pettit Cove (Panel c), and sediments in the Fisherman's Park coves (Panel d), consistent with a common historical source potentially from the former Durez NT facility. These congener TEQ profiles are different from those in upstream regional background locations (Panel a).

### *2.1.2 Biota*

A time series of tissue D/F concentrations in caged mussels deployed by MECP since 1991 is presented in Figure 2-4, including Pettit Cove, Little Niagara River (immediately upstream and downstream of Pettit Cove), and in Fisherman's Park (note that samples in the Fisherman's Park coves were only collected in 2012 and 2015). Like surface sediment, total D/F TEQ[7] concentrations in Pettit Cove mussel tissue have declined roughly 10- to 100-fold over the last 30 years, likely in response to upland source control and remediation activities (Section 1.1). Also, like surface sediment, recent mussel tissue D/F concentrations in Pettit Cove are above regional background levels.[8]

The most recent (2015) mussel tissue D/F congener TEQ profile in Pettit Cove is presented in Figure 2-5 (Panel b). For comparison, average D/F TEQ congener profiles in surface sediments at the upstream Niagara River locations and Pettit Cove are also presented in Figure 2-5 (Panel a). As noted in Section 2.1.1, the congener TEQ profile in Pettit Cove surface sediment is different from upstream regional background sediments. Importantly, the congener TEQ profile in Pettit Cove mussel tissue is different from either sediment profile, suggesting that mussel D/F exposure in Pettit Cove may be from a combination of sources including legacy releases from the Durez NT facility to Pettit Cove sediments as well as ongoing upstream regional background sources.

[6] Note that the bars shown in Figure 2-2 for 2017, 2018, and 2019 represent annual averages of the quarterly surface sediment monitoring data collected at location Cove-3.

[7] Mussel tissue D/F TEQ concentrations based on fish TEFs as reported by MECP.

[8] D/F congener concentrations in caged mussels recently deployed by MECP upstream of Pettit Cove have been below laboratory detection limits, limiting comparisons with human health and wildlife tissue-based screening levels that range from approximately 0.07 to 2.3 ng/kg (wet weight basis).

## 2.2 Preliminary Conceptual Site Model Summary

The evaluations presented in Section 2.1 informed the development of a preliminary conceptual understanding of the origin, fate, transport, and bioaccumulation of D/F within Pettit Cove and the Niagara/Little Niagara River, as follows:

- The Durez NT facility historically released D/F through the PCF to Pettit Cove and potentially to sediments in the Niagara/Little Niagara River downstream of Pettit Cove. The D/F congener TEQ profile associated with historical releases from the Durez NT facility is different from ongoing upstream regional background sources.
- Upland source control and remediation activities at both the Durez NT facility and PCF were completed in 2014 (Section 1.1), resulting in a likely 10- to 100-fold reduction in surface sediment and caged mussel tissue D/F concentrations in Pettit Cove.
- The most recent (2015) mussel tissue D/F congener TEQ profile in Pettit Cove suggests exposure from a combination of sources including legacy releases from the Durez NT facility to Pettit Cove sediments as well as ongoing upstream regional background sources.

This preliminary conceptual site model will be refined using the data collected as part of this Work Plan and summarized in a future Aquatic Assessment Report (see Section 4).

# 3 Sampling and Analysis Plan

## 3.1 Data Quality Objectives

DQOs for this Work Plan are:

- *DQO No. 1: Characterization of Sediment D/F Concentrations.* Sediment cores will be collected from various locations to a depth of 2 feet below mudline to characterize the areal and vertical extent of sediment contamination and to determine if site related contaminants are impacting sediments.
- *DQO No. 2: Assessment of Current Fish and Benthic Invertebrate Community, and Characterization of Tissue D/F Concentrations*. Surveys will be conducted to determine benthic invertebrate and fish species that are present in Pettit Cove and Fisherman's Park Cove. Analysis of representative species collected from those areas (and other areas in the Niagara River) will be conducted to characterize D/F concentrations in tissue to determine if site-related contaminants are impacting fish and wildlife resources.
- *DQO No. 3: Determination of Site-Specific SGV*. Surface sediment will be collected from various locations to characterize site-specific $K_{oc}$s. These data will be used to develop a site-specific SGV consistent with the calculation methodology provided in DEC (2014):[9]

**Equation #**

$$SGV_{OC} = AWQS/GV \ \mu g/L * K_{OC}$$

where:

| | | |
|---|---|---|
| $AWQS/GV$ | = | ambient water quality standard or guidance value |
| $K_{OC}$ | = | organic carbon partitioning coefficient, calculated using measured porewater and bulk sediment D/F concentrations, normalized to organic carbon content (black carbon concentrations provide an independent measure of sequestration potential) |

- *DQO No. 4: Assessment of Source Control Effectiveness and Characterization of Natural Recovery Processes.* Sediment cores will be collected at several locations in Pettit Cove and Fisherman's Park Cove to assess the effectiveness of source control efforts completed to date, and to evaluate physical processes that control the rate of natural recovery in the system (i.e., sedimentation and bioturbation/mixing). The assessment of source control effectiveness

[9] A site-specific SGV will be calculated because research over the last several decades has demonstrated that soot, coal, and charcoal (collectively, "black" carbon) frequently present in urban sediments strongly bind hydrophobic chemicals (including D/F). Literature-based $K_{oc}$ values for D/F do not account for sequestration by black carbon and often significantly underestimate site-specific $K_{oc}$ values (Gustafsson et al. 1996; Luthy et al. 1997; Cornelissen et al. 2005; Lohmann et al. 2005).

will be supplemented by the collection and analysis of sediments that have accumulated in the PCF since the completion of the PCF cleaning activities in 2014.

## 3.2 Sampling Approach

The overall sampling, analysis, and survey DQOs are described in Section 3.1. More detailed descriptions of the scope of work are provided in Section 3.3.

## 3.3 Scope of Work

### *3.3.1 Aquatic Sampling*

#### 3.3.1.1 Porewater Sampling

To address DQO No. 3 and develop a site-specific SGV, concentrations of freely-dissolved D/F in sediment porewater will be measured ex situ using low-density polyethylene (LDPE) passive samplers.[10] For this evaluation, bulk surface sediment samples (approximately 0 to 10 cm) will be collected from three locations in Pettit Cove, two locations in Fisherman's Park Cove, and two locations in the Little Niagara River upstream of Pettit Cove[11] (Figure 3-1). These surface sediment samples will be collected using a Ponar-type grab sampler, placed into the appropriate sample container, and immediately shipped to the Anchor QEA Environmental Geochemical Laboratory in Portland, Oregon for ex situ passive sampler analysis (a standard operating procedure [SOP] for collection and processing of sediment grab samples in support of this study is included in Appendix A). Specifically, LDPE sheets spiked with isotopically labeled (carbon-13 [$^{13}$C]) performance reference compounds (PRCs; i.e., compounds that are used to track equilibration status) will be deployed in each of the bulk sediment samples and allowed to equilibrate for 60 days. A rotary agitator will be used for continuous and aggressive agitation to accelerate the equilibration process. At the end of 60 days, sampling and analysis of the LDPE sheets will be performed in accordance with U.S. Environmental Protection Agency (EPA; 2017), Jonker et al. 2018, and Anchor QEA and Baird (2019). Additional details regarding the SOP for ex situ porewater passive sampling using LDPE is provided in Appendix A.

For this Work Plan, D/F concentrations measured on the LDPE will be converted to freely dissolved porewater D/F concentrations using LDPE-water partition coefficients calculated using published linear free energy relationships (Adams et al. 2007) and octanol-water partition coefficients for D/F (Hawker and Connell 1988) as described in Anchor QEA and Baird (2019).

[10] Research over the last several decades has demonstrated that passive sampling using LDPE in controlled ex situ laboratory environments provides reliable characterization of freely dissolved D/F porewater concentrations suitable for risk assessment and management of contaminated sediments (Jonker et al. 2018).

[11] Note that one of the two upstream locations is located immediately upstream of Pettit Cove coincident with a prior MECP caged mussel survey location.

In addition to analysis of the LDPE, the bulk sediment samples collected to support this porewater evaluation will be analyzed for bulk D/F congeners (dry weight basis), total organic carbon (TOC), and black (soot) carbon.

### 3.3.1.2 Sediment Sampling

#### *3.3.1.2.1 High-Resolution Sediment Coring*

To address DQO No. 4, high-resolution (i.e., finely-segmented) sediment cores will be collected at the same locations in Pettit Cove and Fisherman's Park Cove identified for sediment porewater analysis (three in Pettit Cove and two in Fisherman's Park Cove; see Section 3.3.1.1 and Figure 3-1). At each location, a core of the near-surface sediment column will be collected by pushing to refusal.[12] The top 15 cm (approximately 6 inches) in each core will be segmented into 2-cm intervals (8 samples per core)—all 8 of those 2-cm intervals will be submitted for analysis of bulk D/F (dry weight basis), TOC, and black (soot) carbon. The remainder of each core will be frozen intact and archived until D/F results for the surficial intervals are received and evaluated. If additional deeper finely-segmented (2-cm) intervals are needed to evaluate vertical gradients in D/F concentrations in a given core, those additional samples will be generated (as needed) from the remainder of the core and submitted for analysis. Any remaining sediment will be segmented and analyzed such that samples can be mathematically composited into 6- to 12-inch and 12- to 24-inch concentrations.[13] SOPs related to the collection and processing of the finely-segmented sediment cores is provided in Appendix D.

One additional core will be collected from each of the two locations identified in Fisherman's Park Cove for radioisotope analysis (Cesium-137 [Cs-137] and Lead-210 [Pb-210]) to estimate net sedimentation rates and mixing/bioturbation depths. (*Radioisotope cores will not be collected in Pettit Cove because prior dredging that occurred in this area disturbed the geochronological record*). Analysis of Cs-137 allows for the dating of sediment layers and estimation of deposition rates since the 1950s, due to fallout activity from open-air nuclear testing that was initiated in 1955 and peaked around 1963 (Pennington et al. 1973). Atmospheric Cs-137 fallout was a byproduct of these nuclear tests and Cs-137 data from finely-sectioned sediment cores reflects the historical fallout chronology. Further, analysis of Pb-210 (half-life 22.3 years) allows for a reliable dating of sediment deposited over the last 100 to 150 years, along with surface sediment mixing/bioturbation depths (Krishnaswami et al. 1971). Moreover, Pb-210 dating can also provide information on the rate of sedimentation. For the radionuclide evaluation, the top 2 feet (approximately 60 cm) will be processed into 2-cm intervals (30 samples per core); however, only select intervals will be submitted for radionuclide analysis (i.e., it

[12] Based on recent (May 2019) measurements of sediment thickness conducted by GHD in Pettit Cove, it is anticipated that there is approximately 2 feet (60 cm) of sediment at the target locations in this area. Total sediment thickness in Fisherman's Park Cove is unknown, but is anticipated to be greater than 60 cm.

[13] While the total length of each core that is processed into 2-cm intervals may vary, all sediment cores will have D/F analysis performed to a total depth of 2 feet.

is not necessary to submit all 30 samples from each core to conduct the radionuclide evaluation described above; assumed a maximum of 15 to 20 samples per core).

#### *3.3.1.2.2 Sediment Characterization Sampling*

To address DQO No. 1, sediment cores will be collected from various locations to a depth of 2 feet to characterize the areal and vertical extent of sediment D/F contamination. Cores will be collected from each of five sampling areas, including: 1) Pettit Cove; 2) Fisherman's Park Coves; 3) an area between Pettit Cove and Fisherman's Park Cove (if a sediment deposit can be identified in this area); 4) a depositional area downstream of Fisherman's Park; and 5) an upstream reference area. The high-resolution sediment coring described in Section 3.3.1.2.1 for Pettit Cove and Fisherman's Park Coves meets this objective in those two areas. In the other three areas, two cores will be collected in each area to a total depth of 24 inches (or less if insufficient sediment is present) and processed into 0- to 6-inch, 6- to 12-inch, and 12- to 24-inch samples. Proposed sampling locations are shown in Figure 3-1. It should be noted that because there is some uncertainty regarding the presence/absence of sediment deposits in the area between Pettit Cove and Fisherman's Park, and in the area downstream of Fisherman's Park, this figure shows an approximation of the targeted sampling area. Specific sampling locations may need to be adjusted based on field conditions—any field adjustment to targeted sediment sampling locations will be discussed with DEC. All samples will be analyzed for bulk D/F congeners (dry weight basis), TOC, and black (soot) carbon.

### 3.3.1.3 Fish Community Survey and Tissue Sampling

To address DQO No. 2, a fish community survey will be performed and representative samples of forage and sport fish specimens will be collected for tissue analysis. Fish tissue sample collection will be conducted in the same five general areas described above for sediment characterization sampling, including: 1) Pettit Cove; 2) Fisherman's Park Coves; 3) an area between Pettit Cove and Fisherman's Park Cove; 4) an area downstream of Fisherman's Park; and 5) an upstream reference area[14] (Figure 3-2). Compared with sport fish, forage fish have a relatively small home range; therefore, forage fish species will be collected from all five of these areas. Sport fish including smallmouth bass are typically categorized in bioaccumulation evaluations as "moderately mobile", with a somewhat larger general home range of up to approximately 1 kilometer (km; Gerber 1987; Ettinger-Dietzel et al. 2016; Mycko 2017). Accordingly, sport fish will be collected more broadly from three larger areas to characterize exposure in the Pettit Cove/Fisherman's Park Coves area, the upstream reference area, and an area approximately 1 km downstream of the Fisherman's Park Coves (Figure 3-2).

[14] The upstream reference area (two inlets located opposite the southern tip of Tonawanda Island approximately 1.2 kilometer (km) from Pettit Cove; see Figure 3-2) was selected based on an initial site reconnaissance. The specific location of this upstream reference area may be adjusted in the field in consultation with DEC. This area is expected to be outside of the typical travel range of forage and sport fish to be collected from Pettit Cove (smallmouth bass, bluegill, and bluntnose minnow).

Five composites of forage fish will be collected from each of the 5 forage fish sampling areas depicted in Figure 3-2 (i.e., 25 forage fish samples), and 5 individual sport fish will be collected from the three sport fish areas (15 sport fish samples), for a total of 40 samples. Target forage fish species will be bluegills and bluntnose minnows based on a preliminary understanding of the types of fish that have been observed previously in this area. Approximately 5 bluegill or 25 bluntnose minnows will be required per composite to meet the analytical mass targets for detection limits presented in Table 3-4, though numbers could vary based on the weight of captured fish. Composites will consist of fish of the same species and similar lengths. As discussed in Appendix B, compositing will be by size class (composite samples will be made up of sufficient individuals of similar size (i.e., the smallest individual in a composite will be no less than 75% of the total length of the largest individual).

Target sport fish species will be smallmouth bass based on a preliminary understanding of the types of fish that have been observed previously in this area. However, the overall objective of the sport fish sampling will be to collect the target number of samples (five per sampling area). Sport fish samples may include additional species if insufficient numbers of smallmouth bass are available (yellow perch is proposed as an alternative species based on prior observations). Compositing procedures for sport fish are provided in Appendix B.

Several fish collection methods may be used including both passive and active sample collections methods. Multiple gear allows for a more holistic assessment. Preference will also be given to the use of methods with low mortality rates. Active sample collection methods may include netting fish while wading, netting fish from a boat, and electroshocking. A similar level of effort regarding time spent surveying will be employed in each area and DEC will be consulted in the field to confirm that the sampling effort is appropriate for this Work Plan. Passive sample collections methods would be considered if active methods yield limited fish and may include minnow traps and other types of passive net sampling (for example, hoop nets). Details of all collection and processing methods are described in Appendix B.

Captured fish will be placed in large buckets to reduce mortality rates until field processed. Processing will include species identification, weighing (nearest 1.0 gram for adult sport fish and nearest 0.1 gram for forage species as detailed in Appendix B), measuring total length (in millimeters), and recording any observed external abnormalities. Representative fish may be submitted to a taxonomy laboratory for species verification if required. Collected fish meeting specifications for D/F analysis will be held on ice until the completion of the sampling program and will be submitted for analysis. Fish not needed for chemical analysis will be released. Methods for fish identification, fish measurement, identification of abnormalities, and fish sample archiving methods are described in Appendix B. Anchor QEA will obtain a License to Collect and Possess through DEC prior field implementation and will follow communication requirements set therein.

#### 3.3.1.4 Benthic Invertebrate Community Survey and Tissue Sampling

Also to address DQO No. 2, a benthic community survey will be conducted for mussels and other invertebrates. A focused reconnaissance will be conducted in each of the five areas identified for sediment and fish tissue characterization sampling above to qualitatively evaluate mussel presence and collect specimens for D/F analysis. DEC (2021) guidance will be followed for this survey, and will include boating in the vicinity of, walking, or otherwise accessing hard and soft substrate areas to perform a qualitative visual search of the surface (in-sediment and on surface substrates) for mussels.

In addition, a non-mussel benthic community survey will be conducted in those same five areas to evaluate the presence and species of non-mussel benthic invertebrates within the surficial 24 inches of sediment. These specimens will be collected primarily for identification and enumeration; however, they will be analyzed for D/F if insufficient mussel tissue is able to be collected for analysis (Figure 3-2). It is anticipated that three replicate cores will be needed at each of the proposed locations to obtain sufficient tissue for enumeration and analysis (if needed). Sediment cores collected for benthic invertebrate analysis will be segmented into 6-inch intervals to better characterize the depth of bioactivity at the site.

Details of the benthic community sample sieving methods are described in Appendix C. In summary, sediment push core samples will be sieved in the field to remove excess sediment (silt and fine sand). All invertebrates that are obtained will be submitted for identification and enumeration (up to four taxonomy samples per location). The resulting data will be compiled and analyzed to identify the benthic community that is present (and to what depth those benthic organisms are present) and provide preliminary estimates of diversity, abundance, and dominance at each sampling locations. If it is determined that these non-mussel invertebrates need to be submitted for analysis of D/F, the tissue collected from each area within a single depth interval will be composited for analysis, for a maximum of three samples per area if sufficient mass is obtained (i.e., a single sample for the 0- to 6-, 6- to 12-, and 12- to 24-inch depth intervals will be composited for each of the five sampling areas, for a total of 15 samples).

### *3.3.2 PCF Accumulated Sediment Sampling*

To address DQO No.4, grab samples of sediments that have accumulated in the PCF since it was cleaned in 2014 will be collected and analyzed for D/F. These samples will provide a direct measurement of D/F concentrations in sediments entering Pettit Cove from the PCF. Sediment samples will be collected from three target locations (i.e., manholes) along the PCF in areas where sediments are anticipated to have accumulated since 2014 (based on information collected during the prior clean-out and inspection), including manholes "ROSEBROCK MH-1", "GILMORE MH-1", and "RIVER MH-1" (Figure 3-3). Grab samples of accumulated sediments will be collected using a decontaminated stainless-steel trowel, homogenized, and submitted for analysis of bulk D/F (dry weight basis), TOC, and black (soot) carbon. In addition, one surface sediment grab sample will be

collected in Pettit Cove as close as possible to the PCF outfall using a Ponar-type grab sampler and submitted for analysis.

### *3.3.3 Permitting*

Prior to initiating the work, appropriate permits will be obtained and utility mark outs will be coordinated with Dig Safely New York. The work proposed herein is located within navigable waters of the United States and therefore requires a U.S. Army Corps of Engineers (USACE) permit that will be covered under Nationwide Permit No. 6 Survey Activities. The proposed work also requires a DEC Protection of Waters Permit. USACE and DEC permits will be applied for using New York State's joint permit application and will require consultation with the DEC Division of Fish and Wildlife and the State Historic Preservation Office. It is anticipated that the project would be considered Minor, which streamlines the permitting process.

## 3.4 Sample Custody, Packaging, and Shipping Requirements

Chain-of-custody (COC) procedures will be followed for all samples throughout the collection, handling, and analysis process. The principal document used to track possession and transfer of samples is the COC form. Each sample will be represented on a COC form the day it is collected. COCs can be generated electronically or manually. If manually, data entries will be made using indelible ink pen. Corrections will be made by drawing a single line through the error, writing in the correct information, then dating and initialing the change. Blank lines or spaces on the COC form will be lined out, dated, and initialed by the individual maintaining custody.

A COC form will accompany each cooler of samples sent to the analytical laboratories. Each person who has custody of the samples will sign the COC form and establish that the samples were not left unattended unless properly secured. Copies of COC forms will be retained in the project files.

Filled sample containers for analyses will be stored in coolers containing wet ice to maintain the samples at 0°C to 6°C until delivery to the analytical laboratories.

Samples will be shipped to the analytical laboratory no later than the day after collection, and will be timed to avoid weekend delivery (unless coordination with the laboratory has been performed in advance) so as not to jeopardize any hold time requirements (Table 3-1). Specific sample shipping procedures are as follows:

- Each cooler or container with the samples for analyses will be hand-delivered, couriered, or shipped the same day as collection or via overnight delivery to the appropriate analytical laboratory. If Saturday delivery is required, the field coordinator will contact the analytical laboratory before 3:00 p.m. on Friday to ensure that the laboratory will be staffed to receive samples on a Saturday and is aware of the number of containers shipped and the airbill

tracking numbers for those containers. Following shipment, the field coordinator will confirm the samples have been received and are in good condition.

- Coolant wet ice will be sealed in separate zip-top plastic bags and placed in the shipping containers. Plastic bags with ice will be doubled for overnight shipping.
- Individual sample containers will be placed in a sealable plastic bag, packed to prevent breakage, and transported in a sealed ice chest or other suitable container.
- Glass bottles and jars will be separated in the shipping container by shock-absorbent material (e.g., bubble wrap) to prevent breakage.
- The shipping containers will be wrapped securely with packing tape if being shipped and clearly labeled with sufficient information (name of project, time, and date container was sealed, person sealing the container, and consultant's office name and address) to enable positive identification.
- COC forms will be enclosed in a plastic bag and placed inside of the cooler.
- A minimum of two signed and dated custody seals will be placed on adjacent sides of each cooler prior to shipping. Custody seals are not required when custody is maintained and transferred directly.

Upon transfer of sample possession to the analytical laboratory, the person transferring custody of the sample container will sign the COC forms. Upon receipt of samples at the laboratory, the custody seals will be broken, if applicable, and the receiver will sign the COC forms and record the condition of the samples and any discrepancies encountered on a sample receipt form. Tissue samples collected during community surveys will be stored in plastic bags, wrapped in foil, and labeled prior to shipment. These will be stored in frozen archive at the laboratory pending analyses.

## 3.5 Analytical Methods

Analytical methods and reporting limits are listed in Tables 3-2, 3-3, and 3-4 for the sediment, porewater, and tissue analyses, respectively. SGS North America in Wilmington, North Carolina will analyze sediment, passive sampler material (LDPE), and tissues for D/F; Alpha Analytical in Mansfield, Massachusetts will analyze sediment samples for TOC and soot carbon; and Teledyne-Brown Engineering in Knoxville, Tennessee will analyze sediment samples for Pb-210 and Cs-137. Tissue samples collected during the community surveys may be analyzed for D/F and lipids after the sediment and porewater investigations are complete. A minimum of 50 grams of tissue is required for analyses and tissues can be held for up to 1 year after collection if stored frozen (< 0°C) prior to analyses. In completing analyses for this project, the laboratories are expected to meet the following minimum requirements:

- Adhere to the methods listed in Tables 3-2, 3-3, and 3-4.
- Follow documentation, custody, and sample tracking procedures.
- Notify the Project QA Manager of any QA/QC problems when they are identified.

- Provide a detailed discussion of any modifications made to approved analytical methods.
- Deliver Adobe PDF and electronic data as specified.
- Meet reporting requirements for deliverables.
- Meet turnaround times for deliverables.
- Implement QA/QC procedures, including the DQOs, laboratory QA requirements, and performance evaluation testing requirements.
- Allow laboratory and data audits to be performed, if deemed necessary.

## 3.6 Quality Assurance/Quality Control

Field and laboratory activities must be conducted in such a manner that the results meet specified quality objectives and are fully defensible. Guidance for QA/QC is derived from the protocols developed for EPA's *Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods* (EPA 1986), the EPA National Functional Guidelines for Data Review (EPA 2016a, 2016b), and the cited methods.

### *3.6.1 Field Quality Control*

Field personnel will identify and label samples in a consistent manner to ensure that field samples are traceable, and labels provide the information necessary for the laboratory to properly conduct the required analyses. Samples will be placed in appropriate containers and preserved for shipment to the laboratory.

#### 3.6.1.1 Sample Containers

The analytical laboratories will provide certified pre-cleaned sample containers (Table 3-1). The laboratories will maintain documentation certifying the cleanliness of bottles and the purity of preservatives provided.

#### 3.6.1.2 Sample Identification and Labels

Each sample will have an adhesive plastic or waterproof paper label affixed to the container and will be labeled at the time of collection. The following information will be recorded on the container label:

- Project name
- Sample identification
- Date and time of sample collection
- Preservative type (if applicable)
- Required analyses
- Sampler's name or initials

Samples will be uniquely identified with a sample identification that, at a minimum, specifies sample matrix, sample number, sample location, and type of sample.

#### 3.6.1.3 Field Quality Assurance Sampling

Field QA procedures will consist of following procedures for acceptable practices for collection and handling of samples. Adherence to these procedures will be complemented by periodic and routine equipment inspection.

Field QA samples will be collected along with the environmental samples. Field QA samples are useful in identifying possible problems resulting from sample collection or sample processing in the field. The collection of field QA samples includes equipment rinsate blanks and field duplicates as specified in Table 3-5. Rinsate blanks will be collected at a frequency of one per collection method per sampling event for collection methods that do not use dedicated sampling equipment per location. If decontamination procedures are not adequate, additional rinsate blanks will be collected after procedures have been modified. Adequacy of decontamination procedures will be evaluated by rinsate blank chemistry results. Results will be compared to associated samples, and the Project QA Manager's best professional judgment will be used to evaluate whether decontamination procedures should be modified. Field duplicate samples will be collected at a frequency of one per sampling event or 1 in 20 samples collected, whichever is more frequent.

Field QA samples will also include the collection of additional sample volume or mass to ensure that the laboratory has a sufficient sample amount to analyze the method and program-required analytical QA/QC (matrix duplicate/matrix spike [MD/MS]) samples as specified in Table 3-5. Additional sample volume or mass to meet this requirement will be collected at a frequency of one per sampling event or 1 in 20 samples processed, whichever is more frequent. The sample collection team will confirm with the laboratory the appropriate extra volume or mass required for these analyses. The samples designated for MD/MS analyses should be clearly marked on the COC form.

Field QA samples will be documented on the field forms and verified by the Project QA Manager or designee.

### *3.6.2 Chemistry Laboratory Quality Control*

Laboratory QC procedures, where applicable, include initial and continuing instrument calibrations, standard reference materials, laboratory control samples (LCSs), matrix replicates, MSs, surrogate spikes (for organic analyses), and method blanks. Table 3-5 lists the frequency of analyses for laboratory QA/QC samples, and Table 3-6 summarizes the DQOs for precision, accuracy, and completeness.

An analyst will review the results of the QC samples from each analytical batch immediately after a sample group has been analyzed. The QC sample results will then be evaluated to determine if

control limits have been exceeded. If control limits are exceeded in the batch and reanalysis or re-extraction does not correct the exceedance, the Project QA Manager will be contacted, and alternative corrective action (e.g., method modifications followed by reprocessing the affected samples) will be explored prior to reporting the results.

#### 3.6.2.1 Laboratory Instrument Calibration and Frequency

An initial calibration will be performed on each laboratory instrument to be used prior to analyses, after each major interruption to the analytical instrument, and when any ongoing calibration does not meet method criteria. A calibration verification sample will be analyzed following each initial calibration as required and will meet method criteria prior to analyses of samples. Continuing calibration verifications (CCVs) will be analyzed at required frequencies to track instrument performance. The frequency of CCVs varies with method. For dioxin/furan analyses, one will be analyzed every 12 hours. For TOC and soot carbon methods, one will be analyzed every 10 samples and at the end of each run. CCVs are not analyzed in association with radiochemistry analyses. If the CCV is out of control, the analyses must come to a halt until the source of the failure is eliminated or reduced enough to meet control specifications. Project samples analyzed while instrument calibration is out of control will be reanalyzed.

Instrument blanks or continuing calibration blanks provide information on the stability of the baseline established. Continuing calibration blanks will be analyzed immediately prior to or right after the CCV as applicable to the method.

#### 3.6.2.2 Standard Reference Materials

A standard reference material (SRM) sample will be analyzed in association with the sediment and tissue samples. SRMs will be analyzed with every batch of 20 samples or less and results will be compared to certified and reference values. SRMs will be sourced from known and reliable vendors (e.g., NIST, Cambridge Isotopes) and which ones will be used will be determined prior to project commencement and based on applicability and availability.

#### 3.6.2.3 Laboratory Duplicates

Laboratory duplicates provide information on the precision of the analysis and are useful in assessing potential sample heterogeneity and matrix effects. Laboratory duplicates are subsamples of the original sample that are prepared and analyzed as a separate sample. Laboratory duplicates will be analyzed to assess laboratory precision for all methods. A matrix spike duplicate (MSD), ongoing precision and recovery sample (OPR) duplicate, or LCS duplicate, may be analyzed in lieu of a laboratory duplicate.

#### 3.6.2.4 Matrix Spikes and Matrix Spike Duplicates

Analyses of MS samples provide information on the extraction efficiency of the method on the sample matrix, as well as any interferences introduced by the sample matrix. By performing duplicate MS analyses, information on the precision of the method is also provided. MS/MSDs will be analyzed in association with the TOC, soot carbon, and radiochemistry analyses. See Table 3-5 for required field and laboratory quality control sample analyses and frequencies.

#### 3.6.2.5 Method Blanks

Method blanks are analyzed to assess possible laboratory contamination at every stage of sample preparation and analysis. The method blank results must be less than the reporting limit of each target analyte. If a laboratory method blank exceeds this criterion for any analyte, and the analyte is detected in any of the samples and is less than five times the concentration found in the blank (10 times for common contaminants), analyses must either stop or samples be reanalyzed after the source of contamination is eliminated or reduced.

#### 3.6.2.6 Laboratory Control and Ongoing Precision and Recovery Samples

LCSs and OPRs are analyzed to assess possible laboratory bias at all stages of sample preparation and analysis. The LCS is a matrix-dependent spiked sample prepared at the time of sample extraction along with the preparation of the sample, method blank, and MS. The LCS and OPR will provide information on the accuracy of the analytical process and, when analyzed in duplicate, will provide precision information as well.

#### 3.6.2.7 Laboratory Deliverables

Level 4 data packages will be provided by the laboratories and checked for completeness immediately upon receipt to ensure that requested data and QA/QC information are present. Laboratories will also submit electronic data deliverables in the specified format.

## 3.7 Documentation, Recordkeeping, and Reporting Requirements

GHD currently employs a data management system with a centralized database to manage all of the data collected to date for this site. Therefore, field data and laboratory analytical data generated during this aquatic assessment will be stored in the same project database and managed by GHD following the previously established practices. Anchor QEA will collect field data using tools and applications provided by GHD and will transmit the data to the project database following instructions from GHD. Laboratories will provide analytical data in the electronic data deliverable (EDD) format required by GHD and follow the established process to submit analytical results to the project database.

Original field data documents (e.g., field sheets and field logbooks) will be archived in Anchor QEA's hardcopy project file storage facility. Electronic files (e.g., field data collection applications, EDDs,

electronic data logger files, and photographs) will be submitted to GHD and will be archived and retained following GHD's standard practices.

### *3.7.1 Data Validation and Usability*

Laboratory data will be provided in both PDF and electronic format. Once data are received from the laboratory, QC procedures will be followed to provide an accurate evaluation of the data quality. Stage 4 validations will be conducted in accordance with the EPA National Functional Guidelines for Data Review (EPA 2016a, 2016b) project specific DQOs (Table 3-6), analytical method criteria, and the laboratory's internal performance standards based on their SOPs. During the validation process, analytical data will be evaluated for method and laboratory QC compliance, and their validity and applicability for program purposes will be determined. Based on the findings of the validation process, data validation qualifiers may be assigned. The validated project data, including qualifiers, will be entered into the project database, thus enabling this information to be retained or retrieved as needed. Chemical data will be reviewed with regard to the following, as appropriate to the particular analysis:

- Data completeness
- Holding times
- Instrument performance checks
- Initial calibrations
- Continuing calibrations
- Equipment blanks
- Method blanks
- Labeled standard recoveries
- Detection limits
- Reporting limits
- Laboratory control/ongoing precision and recovery samples
- Field and laboratory duplicates
- MD/MS/MSD samples
- Standard reference material samples
- Recalculation of instrument and sample results
- Instrument output review

The results of the data validation, including text assigning qualifiers in accordance with the EPA National Functional Guidelines for Data Review (EPA 2016a, 2016b) and a tabular summary of qualifiers, will be generated by the validator and submitted to the Project QA Manager for final review and confirmation of the validity of the data. Results of the data validation will be summarized in a Data Usability Summary Report.

## 3.8 Health and Safety

Prior to commencement of the sampling/surveys described in this Work Plan, Anchor QEA will prepare a site-specific health and safety plan, including a COVID-19 Management Plan. In addition, because the field activities described herein require working within the Niagara River, a site-specific job safety analysis (JSA) identifying potential hazards associated with this aquatic field work will be prepared.

# 4 Schedule and Reporting

Anchor QEA anticipates initiating field work within 60 days after DEC approval of this Work Plan. DEC will be notified 2 weeks prior to mobilization for any sampling or field surveys and following notification requirements of the Licenses to Collect and Possess.

The raw (unvalidated) analytical data packages from each of the sampling elements described in this Work Plan will be provided to DEC within 2 weeks of data package delivery to Anchor QEA.

Following completion of all of the field work described in this Work Plan, an Aquatic Assessment Report will be prepared that includes the following: 1) a summary of the validated analytical data results (sediment, porewater, and tissue); 2) a summary of the benthic and fish community survey results; and 3) interpretation, findings, conclusions, and recommendations based on the data collected in this Work Plan, including a proposed site-specific SGV. Also, if the data collected pursuant to this Work Plan suggest that additional data are needed to complete characterization of the nature and extent of sediment D/F concentrations and/or associated environmental risks in Pettit Cove and the Niagara/Little Niagara River, the Aquatic Assessment Report will include recommendations regarding any additional proposed sediment and/or tissue sampling. The Aquatic Assessment Report will be submitted to DEC within 90 days after receipt and completion of validation of all analytical results.

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# Tables

**Table 3-1**
**Sample Sizes, Holding Times, and Preservation**

| Parameter | Sample Size | Container Size and Type | Holding Time | Sample Preservation Technique |
|---|---|---|---|---|
| **Sediment** | | | | |
| Total solid/TOC/soot carbon | 20 g | 8-oz glass | 28 days | < 6°C |
| | | | 6 months | < -10°C |
| Dioxin/furans | 10 g | 4-oz amber glass | 1 year to extraction/ 1 year to analysis | < -10°C |
| Radiochemistry | 100 g | 4-oz glass | None | Ambient |
| **Porewater**[1] | | | | |
| Dioxin/furan | 1 L (sediment) | 2 x 32-oz glass | None established | 2°C–6°C |
| | PE Sampler | 4-oz glass | | |
| **Tissue** | | | | |
| Total solids | 10 g (30 g preferred) | Whole body and fillet: wrap in foil and bag; homogenized tissue: 4-oz amber glass jar | None established | < -10°C |
| Lipids | 10 g (30 g preferred) | | None established | < -10°C |
| Dioxin/furans | 10 g (30 g preferred) | | 1 year to extraction/ 1 year to analysis | < -10°C |

Notes:
1. Sediment will be sent to the EGL for ex situ PE sampler preparation. Once prepared, the PE sample will be sent to SGS for analysis.
g: gram
N/A: not applicable
oz: ounce
PE: polyethylene
TOC: total organic carbon

**Table 3-2**
**Sediment Analytes, Methods, and Target Reporting Limits**

| Parameter | Analytical Method | MDL | MRL |
|---|---|---|---|
| **Conventionals (%)** | | | |
| Total solids | SM 2540G | 0.1 | 0.1 |
| Total organic carbon | 9060A | 0.01 | 0.01 |
| Soot carbon | 9060A Modified | 0.01 | 0.01 |
| **Dioxin/furans (ng/kg)** | | | |
| 2,3,7,8-TCDD | 1613B | 0.17 | 0.5 |
| 1,2,3,7,8-PeCDD | 1613B | 0.51 | 2.5 |
| 1,2,3,4,7,8-HxCDD | 1613B | 0.39 | 2.5 |
| 1,2,3,6,7,8-HxCDD | 1613B | 0.53 | 2.5 |
| 1,2,3,7,8,9-HxCDD | 1613B | 0.45 | 2.5 |
| 1,2,3,4,6,7,8-HpCDD | 1613B | 1.2 | 2.5 |
| OCDD | 1613B | 2.2 | 5.0 |
| 2,3,7,8-TCDF | 1613B | 0.094 | 0.5 |
| 1,2,3,7,8-PeCDF | 1613B | 0.47 | 2.5 |
| 2,3,4,7,8-PeCDF | 1613B | 0.44 | 2.5 |
| 1,2,3,4,7,8-HxCDF | 1613B | 0.27 | 2.5 |
| 1,2,3,6,7,8-HxCDF | 1613B | 0.39 | 2.5 |
| 1,2,3,7,8,9-HxCDF | 1613B | 0.72 | 2.5 |
| 2,3,4,6,7,8-HxCDF | 1613B | 0.40 | 2.5 |
| 1,2,3,4,6,7,8-HpCDF | 1613B | 0.93 | 2.5 |
| 1,2,3,4,7,8,9-HpCDF | 1613B | 0.45 | 2.5 |
| OCDF | 1613B | 2.1 | 5.0 |
| **Radiochemistry (pCi/g)** | | | |
| Lead-210 | TBE-15 | 0.1 | 0.1 |
| Cesium-137 | 901.1 | 0.1 | 0.1 |

Notes:
pCi/g: picocuries per gram
MDL: method detection limit
mg/kg: milligram per kilogram
MRL: method reporting limit
ng/kg: nanogram per kilogram

**Table 3-3**

**Porewater Analytes, Methods, and Target Reporting Limits**

| Parameter | Analytical Method | MDL[1] | MRL[2] |
|---|---|---|---|
| **Dioxin/furans (pg)** | | | |
| 2,3,7,8-TCDD | 1613B | -- | 5.0 |
| 1,2,3,7,8-PeCDD | 1613B | -- | 25 |
| 1,2,3,4,7,8-HxCDD | 1613B | -- | 25 |
| 1,2,3,6,7,8-HxCDD | 1613B | -- | 25 |
| 1,2,3,7,8,9-HxCDD | 1613B | -- | 25 |
| 1,2,3,4,6,7,8-HpCDD | 1613B | -- | 25 |
| OCDD | 1613B | -- | 50 |
| 2,3,7,8-TCDF | 1613B | -- | 5.0 |
| 1,2,3,7,8-PeCDF | 1613B | -- | 25 |
| 2,3,4,7,8-PeCDF | 1613B | -- | 25 |
| 1,2,3,4,7,8-HxCDF | 1613B | -- | 25 |
| 1,2,3,6,7,8-HxCDF | 1613B | -- | 25 |
| 1,2,3,7,8,9-HxCDF | 1613B | -- | 25 |
| 2,3,4,6,7,8-HxCDF | 1613B | -- | 25 |
| 1,2,3,4,6,7,8-HpCDF | 1613B | -- | 25 |
| 1,2,3,4,7,8,9-HpCDF | 1613B | -- | 25 |
| OCDF | 1613B | -- | 50 |

Notes:

1. MDLs are not available for this matrix.
2. MRLs listed are based on analysis of 100% of extract. If split for archival, MRLs will be doubled.

MDL: method detection limit

MRL: method reporting limit

pg: picogram

**Table 3-4**

**Tissue Analytes, Methods, and Target Reporting Limits**

| Parameter | Analytical Method | MDL | MRL |
|---|---|---|---|
| **Conventionals (%)** | | | |
| Lipids | SGS SOP | 0.1 | 0.1 |
| **Dioxin/furans (ng/kg)** | | | |
| 2,3,7,8-TCDD | 1613B | 0.14 | 0.50 |
| 1,2,3,7,8-PeCDD | 1613B | 0.18 | 2.5 |
| 1,2,3,4,7,8-HxCDD | 1613B | 0.23 | 2.5 |
| 1,2,3,6,7,8-HxCDD | 1613B | 0.20 | 2.5 |
| 1,2,3,7,8,9-HxCDD | 1613B | 0.40 | 2.5 |
| 1,2,3,4,6,7,8-HpCDD | 1613B | 0.50 | 2.5 |
| OCDD | 1613B | 2.89 | 5.0 |
| 2,3,7,8-TCDF | 1613B | 0.11 | 0.50 |
| 1,2,3,7,8-PeCDF | 1613B | 0.23 | 2.5 |
| 2,3,4,7,8-PeCDF | 1613B | 0.11 | 2.5 |
| 1,2,3,4,7,8-HxCDF | 1613B | 0.13 | 2.5 |
| 1,2,3,6,7,8-HxCDF | 1613B | 0.18 | 2.5 |
| 1,2,3,7,8,9-HxCDF | 1613B | 0.48 | 2.5 |
| 2,3,4,6,7,8-HxCDF | 1613B | 0.11 | 2.5 |
| 1,2,3,4,6,7,8-HpCDF | 1613B | 0.23 | 2.5 |
| 1,2,3,4,7,8,9-HpCDF | 1613B | 0.84 | 2.5 |
| OCDF | 1613B | 0.82 | 5.0 |

Notes:

MDL: method detection limit

MRL: method reporting limit

ng/kg: nanogram per kilogram

SGS: SGS North America

SOP: standard operating procedure

**Table 3-5**
**Field and Laboratory Quality Control Sample Analysis Frequency**

| Analysis Type | Rinsate Blanks | Field Duplicates | Initial Calibration[1] | Ongoing Calibration | LCS/OPR | Laboratory Duplicates | Matrix Spikes | Method Blanks | Surrogate Spikes |
|---|---|---|---|---|---|---|---|---|---|
| Total solids | N/A | 1 per 20 samples | Daily[2] | N/A | N/A | 1 per 20 samples | N/A | N/A | N/A |
| TOC and soot carbon | N/A | 1 per 20 samples | Daily | Every 10 samples | 1 per 20 samples | 1 per 20 samples | 1 per 20 samples | 1 per 20 samples | N/A |
| Dioxin/furans | 1 per sampling event | 1 per 20 samples | As needed[3] | Every 12 hours | 1 per 20 samples | 1 per 20 samples | N/A[4] | 1 per 20 samples | Every sample |
| Radiochemistry | N/A | N/A | Daily (gamma) Monthly (beta) | N/A | 1 per 20 samples | 1 per 20 samples | 1 per 20 samples | 1 per 20 samples | N/A |

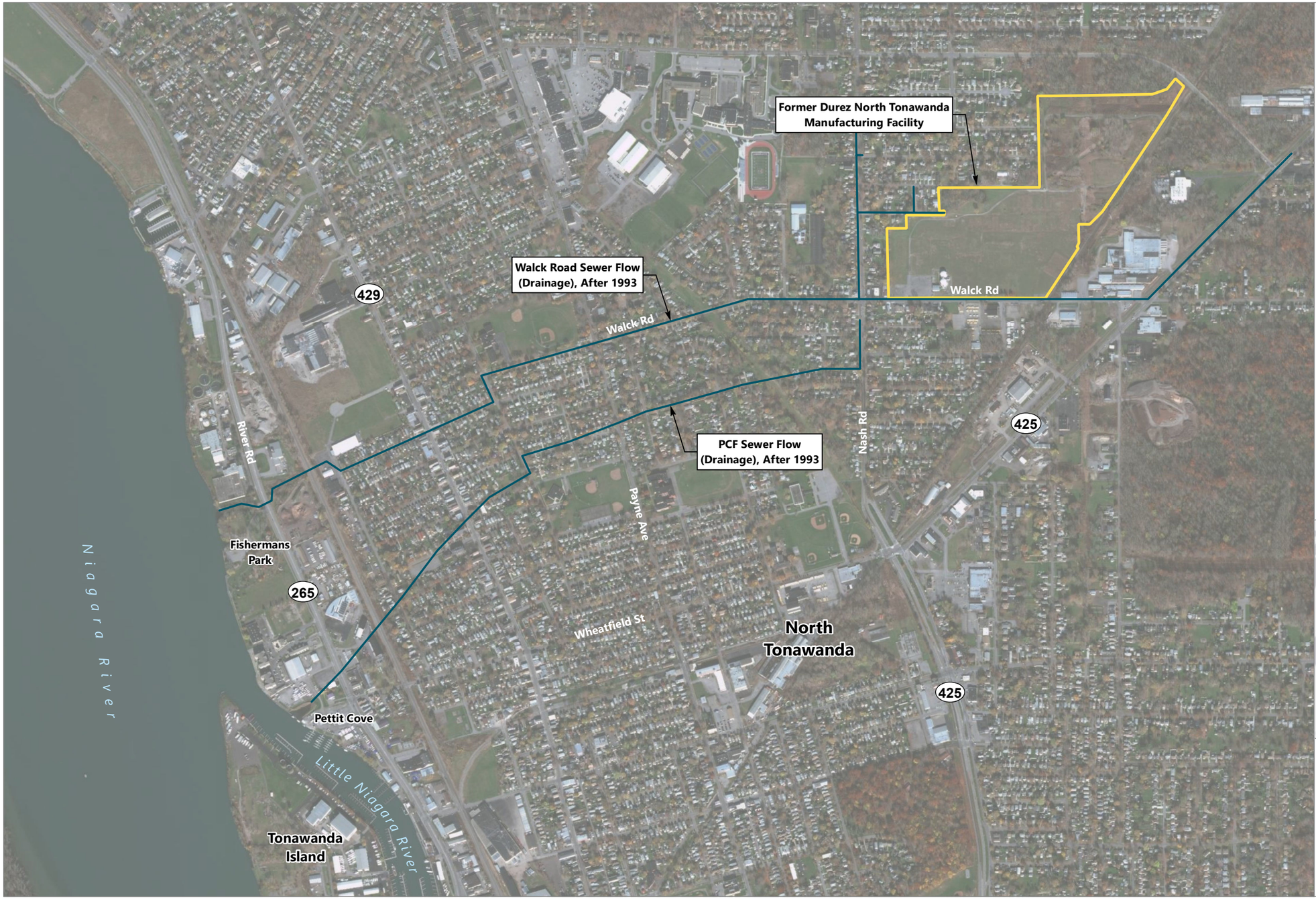
Notes:
1. Initial calibration verification and calibration blank must be analyzed after initial calibration and before samples are analyzed.
2. Calibration and certification of drying ovens and weighing scales are conducted bi-annually.
3. Initial calibrations are considered valid until the ongoing continuing calibration no longer meets method specifications. At that point, a new initial calibration is performed.
4. Isotope dilution method-labeled standards are spiked in every dioxin/furan sample to assess method performance in the sample matrix.
N/A: not applicable
LCS: laboratory control sample
OPR: ongoing precision and recovery sample (used for dioxin/furan analysis)
TOC: total organic carbon

**Table 3-6**
**Data Quality Criteria**

| Parameter | Precision | Accuracy[1] | Completeness |
|---|---|---|---|
| Total solids | ± 20% RPD | N/A | 95% |
| TOC and soot carbon | ± 25% RPD | 75% R – 125% R | 95% |
| Dioxin/furans | ± 30% RPD | 70% R – 130% R | 95% |
| Radiochemistry | ± 30% RPD | 70% R – 130% R | 95% |

Notes:
1. Accuracy goals apply to laboratory control samples and matrix spike samples, as applicable to the analysis.
N/A: not applicable
R: recovery
RPD: relative percent difference
TOC: total organic carbon

# Figures




**LEGEND:**

- Sewer Flow (Drainage), After 1993
- Former Durez North Tonawanda Manufacturing Facility

**NOTE:**
1. Aerial imagery sourced from Esri streaming service, November 2020.

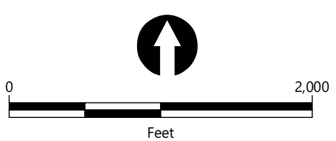




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**Figure 1-1**
**Site Location Map**
Aquatic Assessment Work Plan
Durez Inlet


**LEGEND:**

- GSH Sampling Location
- MECP Sampling Location

**NOTE:**
1. Aerial imagery sourced from Esri streaming service, November 2020.

0 400
Feet

Publish Date: 2021/04/13, 3:58 PM | User: adowell
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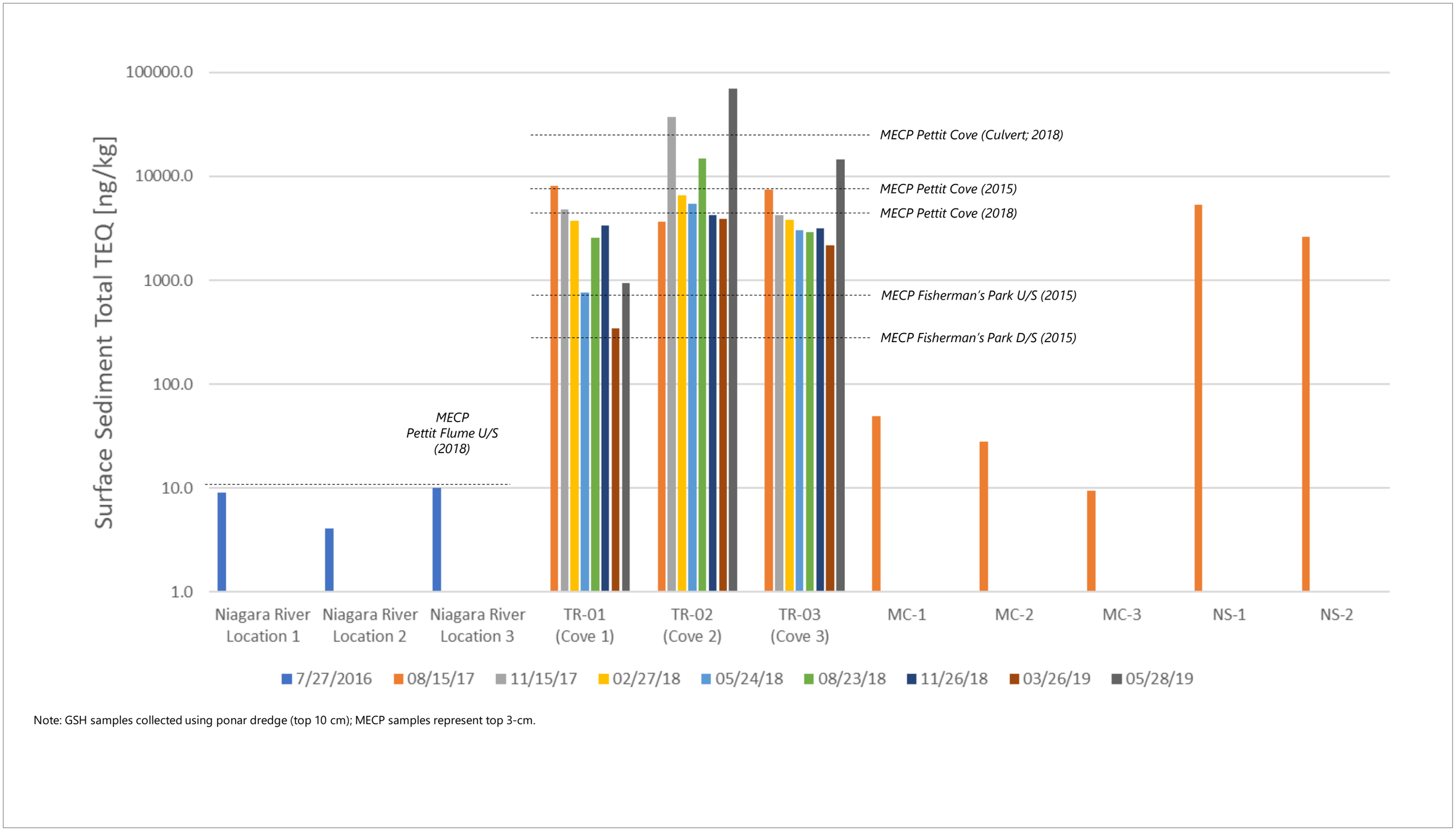
ANCHOR QEA

**Figure 1-2**
**Existing Aquatic Sediment and Biota Sampling Locations (Post-2014)**
Aquatic Assessment Work Plan
Durez Inlet




Note: GSH samples collected using ponar dredge (top 10 cm); MECP samples represent top 3-cm.

Filepath: https://anchorqea-my.sharepoint.com/:b:/r/personal/mwerth_anchorqea_com/Documents/Oxy%20-%20Durez/Aquatic%20Assessment%20Work%20Plan/Figures/Source%20Files/

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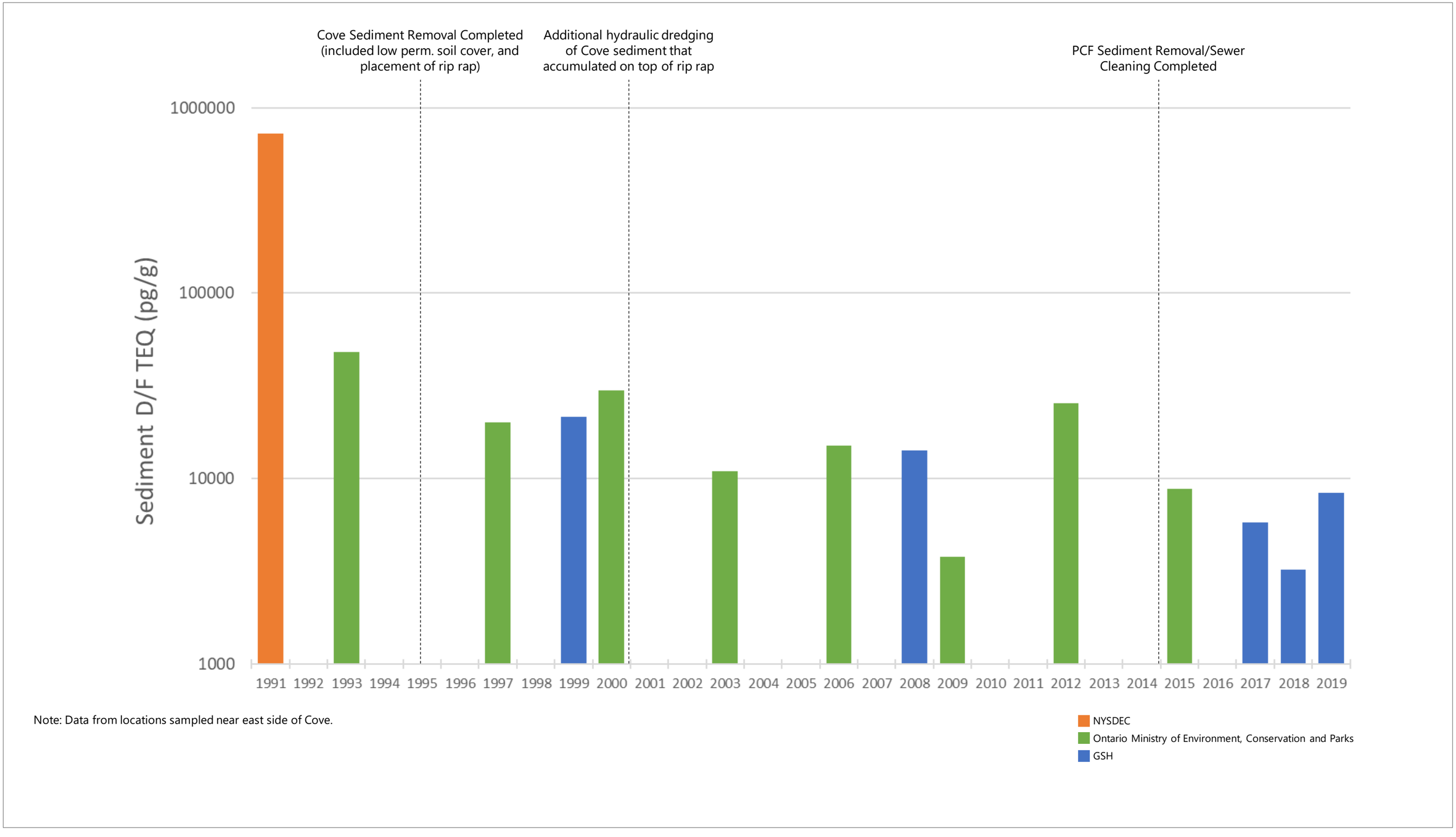
**Figure 2-1**
**Total TEQ Concentrations in Surface Sediment (0 to 10 cm) within Little Niagara River and Pettit Cove**
Aquatic Assessment Work Plan
Durez Inlet




Note: Data from locations sampled near east side of Cove.

Filepath: https://anchorqea-my.sharepoint.com/:b:/r/personal/mwerth_anchorqea_com/Documents/Oxy%20-%20Durez/Aquatic%20Assessment%20Work%20Plan/Figures/Source%20Files/

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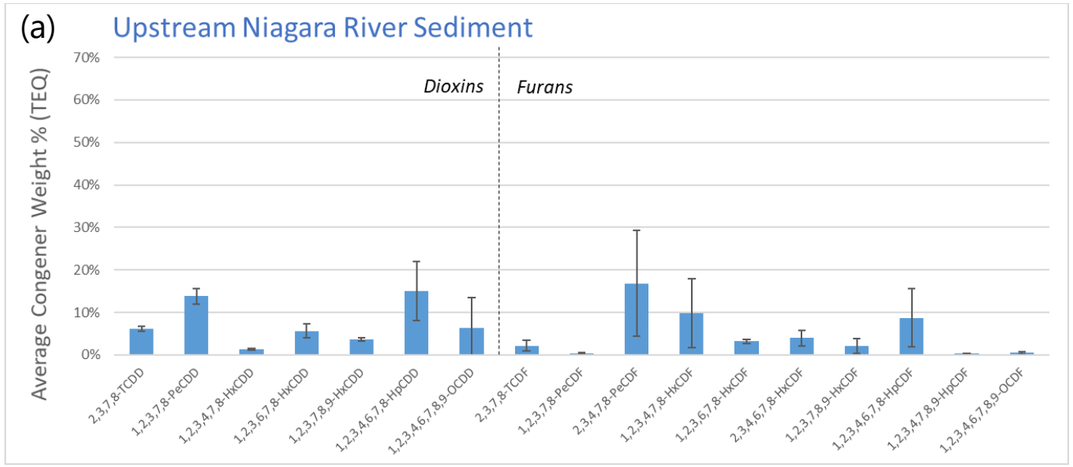
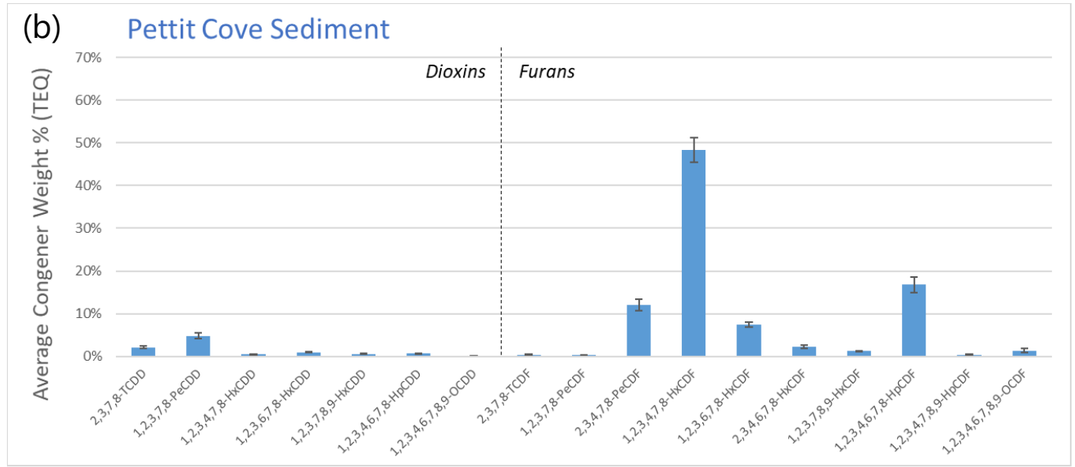
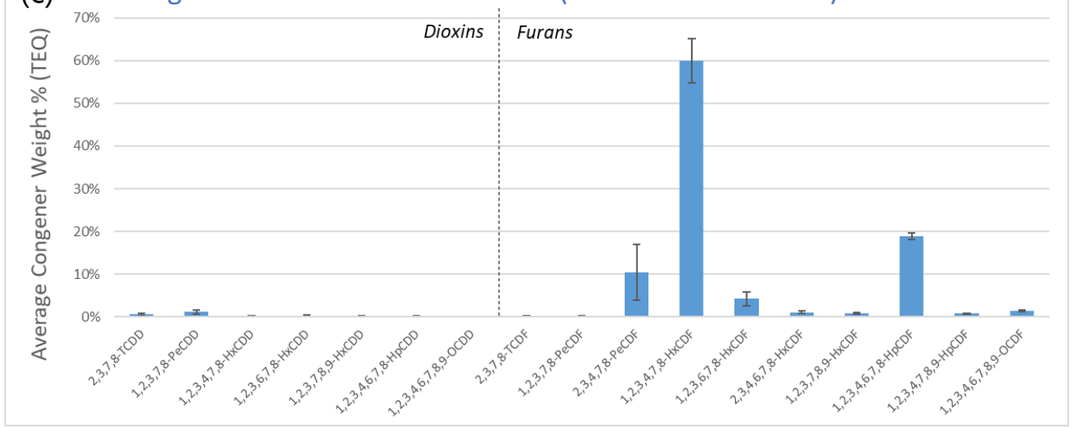
**Figure 2-2**
**Time Series of Surface Sediment Total TEQ in Pettit Cove**
Aquatic Assessment Work Plan
Durez Inlet



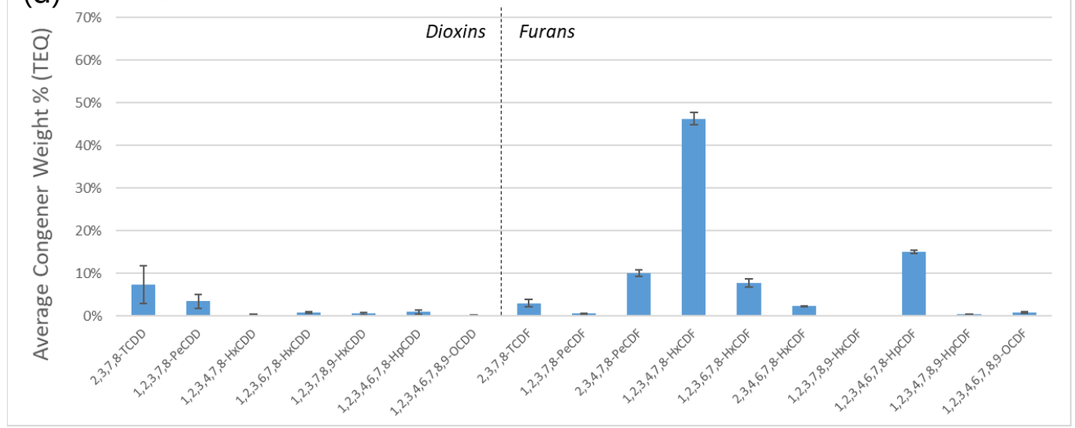




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**Figure 2-3**
**Average Surface Sediment D/F TEQ Congener Profiles**
Aquatic Assessment Work Plan
Durez Inlet

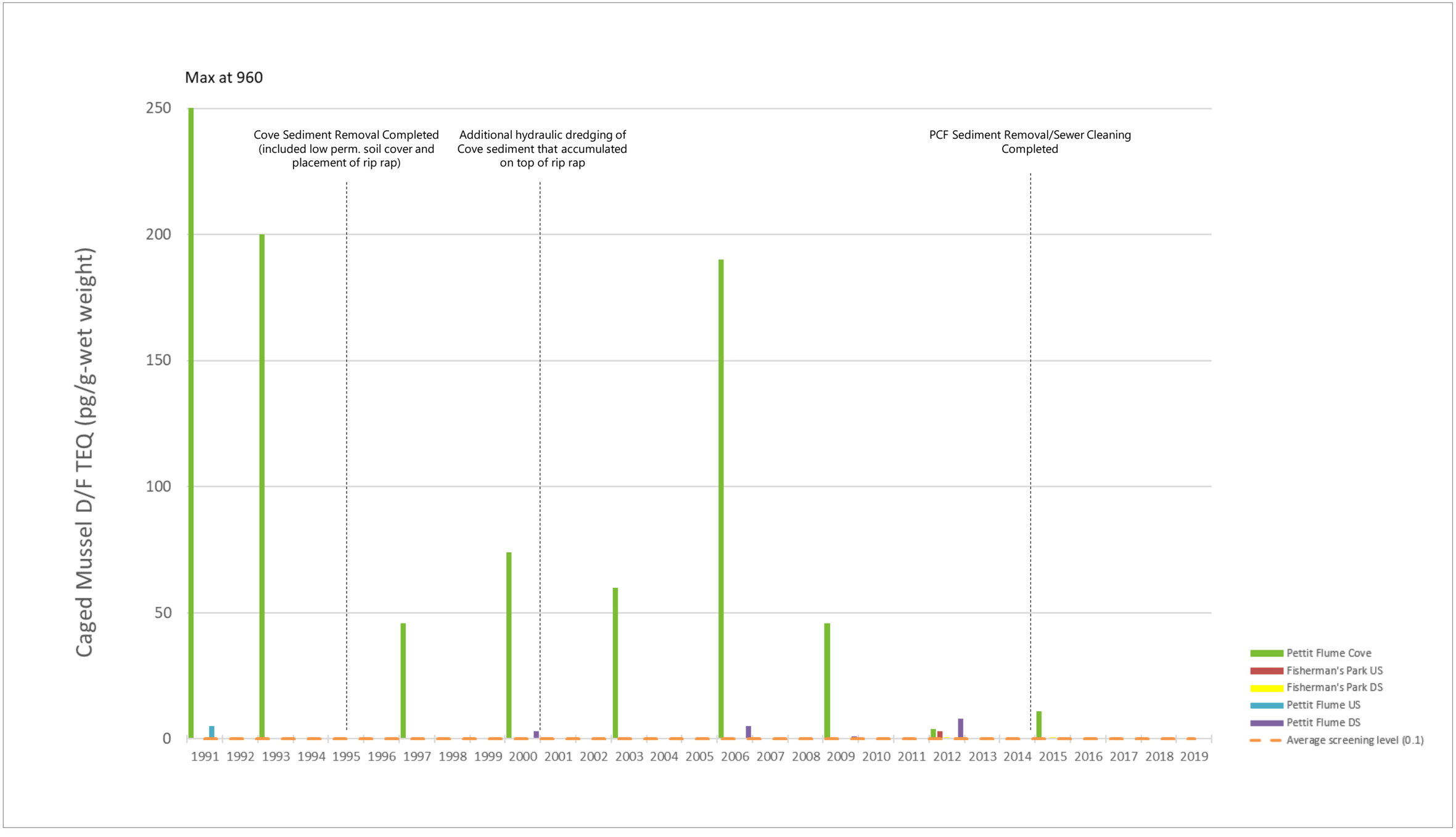


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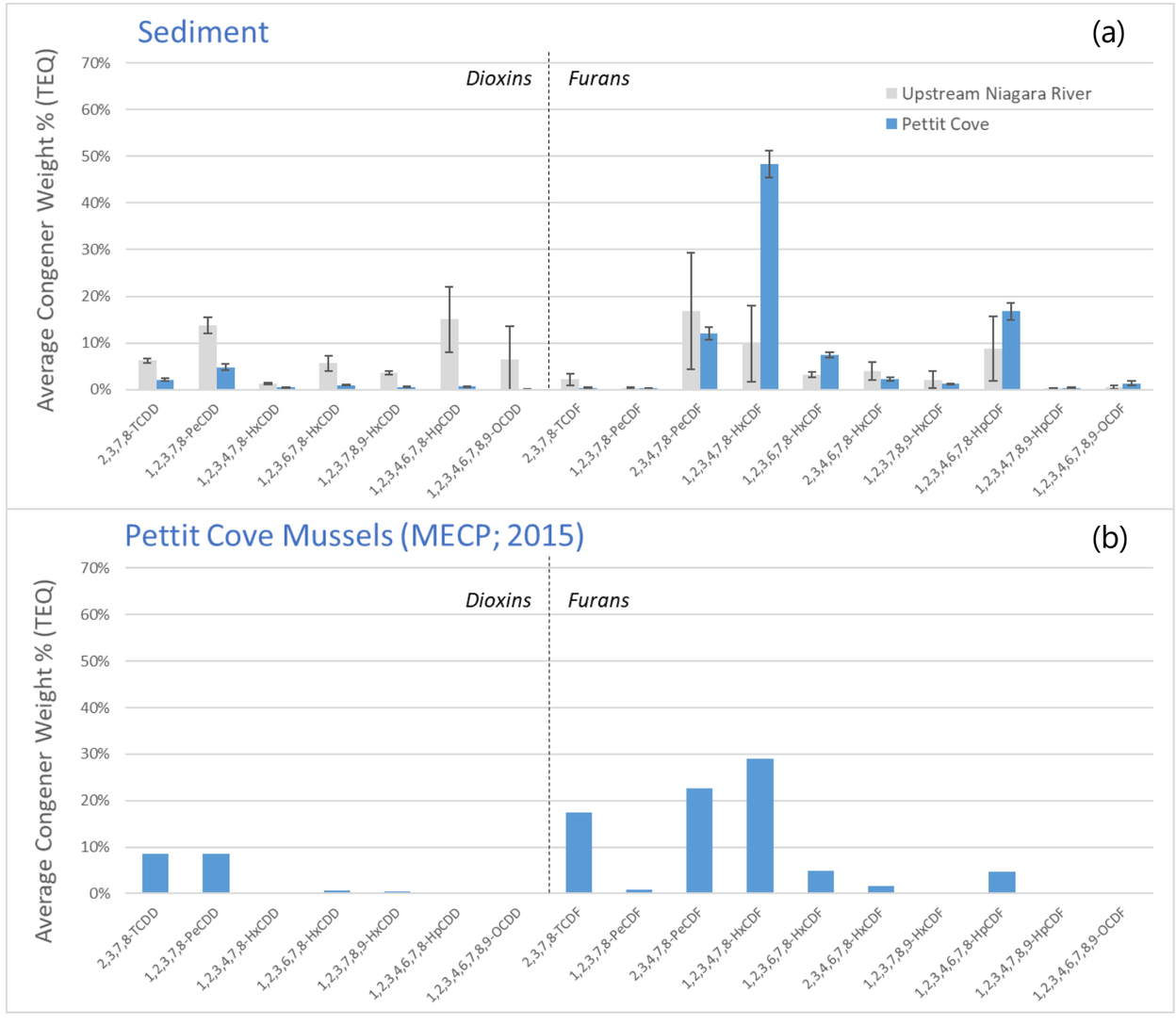
**Figure 2-4**
**Time Series of Total TEQ in MECP Caged Mussels**
Aquatic Assessment Work Plan
Durez Inlet




Notes:
Sediments in Pettit Cove represent average (+/- 2 standard errors) of all Cove sediment samples from three quarterly monitoring locations (TR-01, TR-02, & TR-03; 2017-2019).
Upstream Niagara River sediments represent average (+/- 2 standard errors) of three upstream Niagara River locations (2016).

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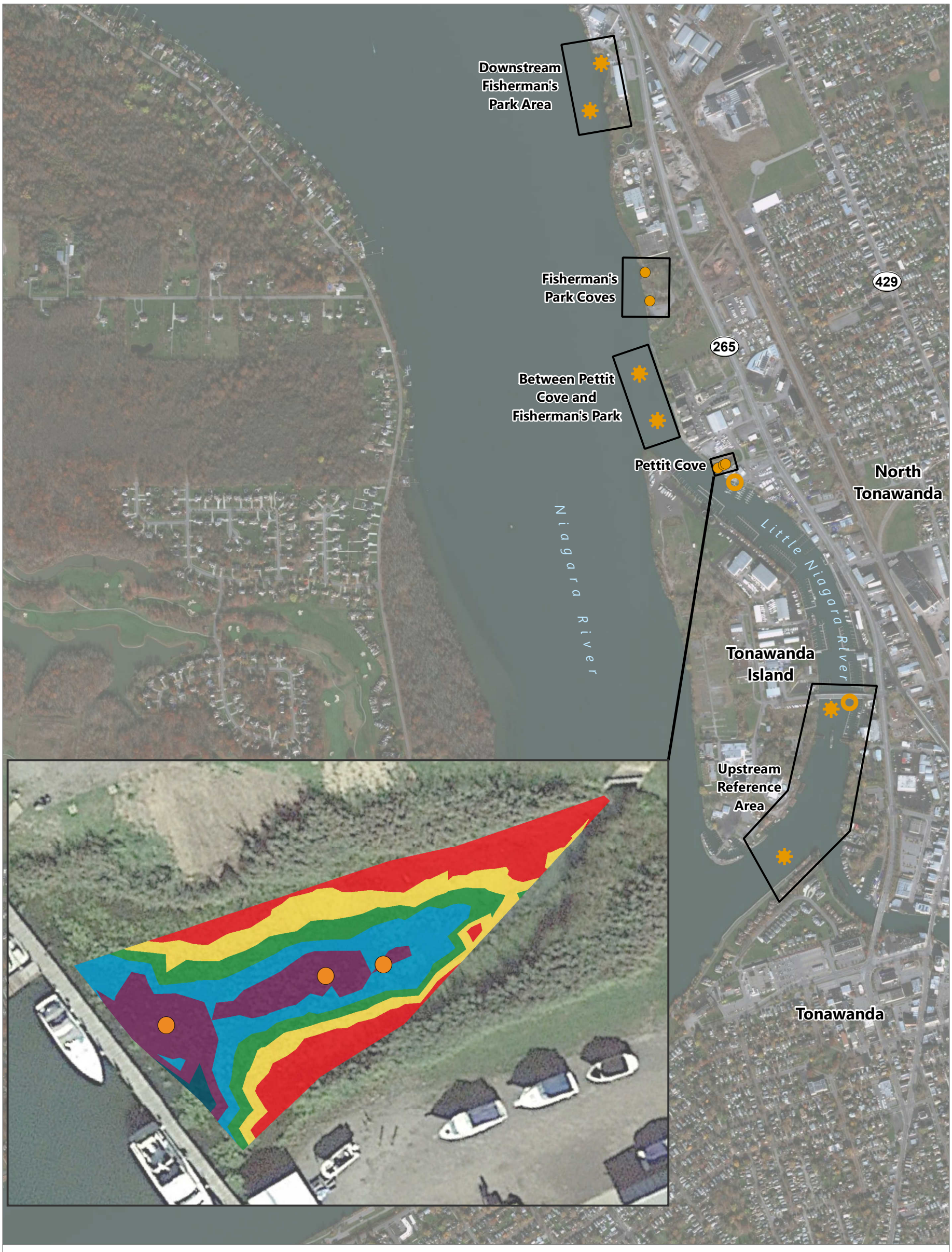


**LEGEND:**

- Targeted Sediment Sampling Area
- Porewater Sampling Location Only
- Porewater and High Resolution Core Sampling Location
- Additional Sediment Characterization Sampling

May 2019 Sediment Thickness Survey in Pettit Cove

| THICKNESS TABLE | | |
|---|---|---|
| MINIMUM THICKNESS | MAXIMUM THICKNESS | COLOR |
| 0.000 | 0.500 | |
| 0.500 | 1.000 | |
| 1.000 | 1.500 | |
| 1.500 | 2.000 | |
| 2.000 | 2.750 | |
| 2.750 | 3.500 | |

**NOTE:**
1. Aerial imagery sourced from Google Earth Pro, September 2018 and Esri streaming service, November 2020.
2. Sediment thickness in feet.

0 1,000
Feet

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**Figure 3-1**
**Sediment Characterization, Porewater, and High Resolution Core Sampling Locations**
Aquatic Assessment Work Plan
Durez Inlet

DRAFT


**LEGEND:**

- Non-mussel benthic invertebrate sampling location
- Forage fish and mussel survey area
- Sport fish area

**NOTES:**
1. Aerial imagery sourced from Esri streaming service, November 2020.

0 2,000
Feet

Publish Date: 2021/07/13, 2:18 PM | User: jquinley
Filepath: \\orcas\GIS\Jobs\Glenn_Springs_Holdings_0049\Oxy_Durez_Inlet\Maps\Aquatic_Assessment_Work_Plan\AQ_Fig3-2_Fish_Community_Survey_L.mxd

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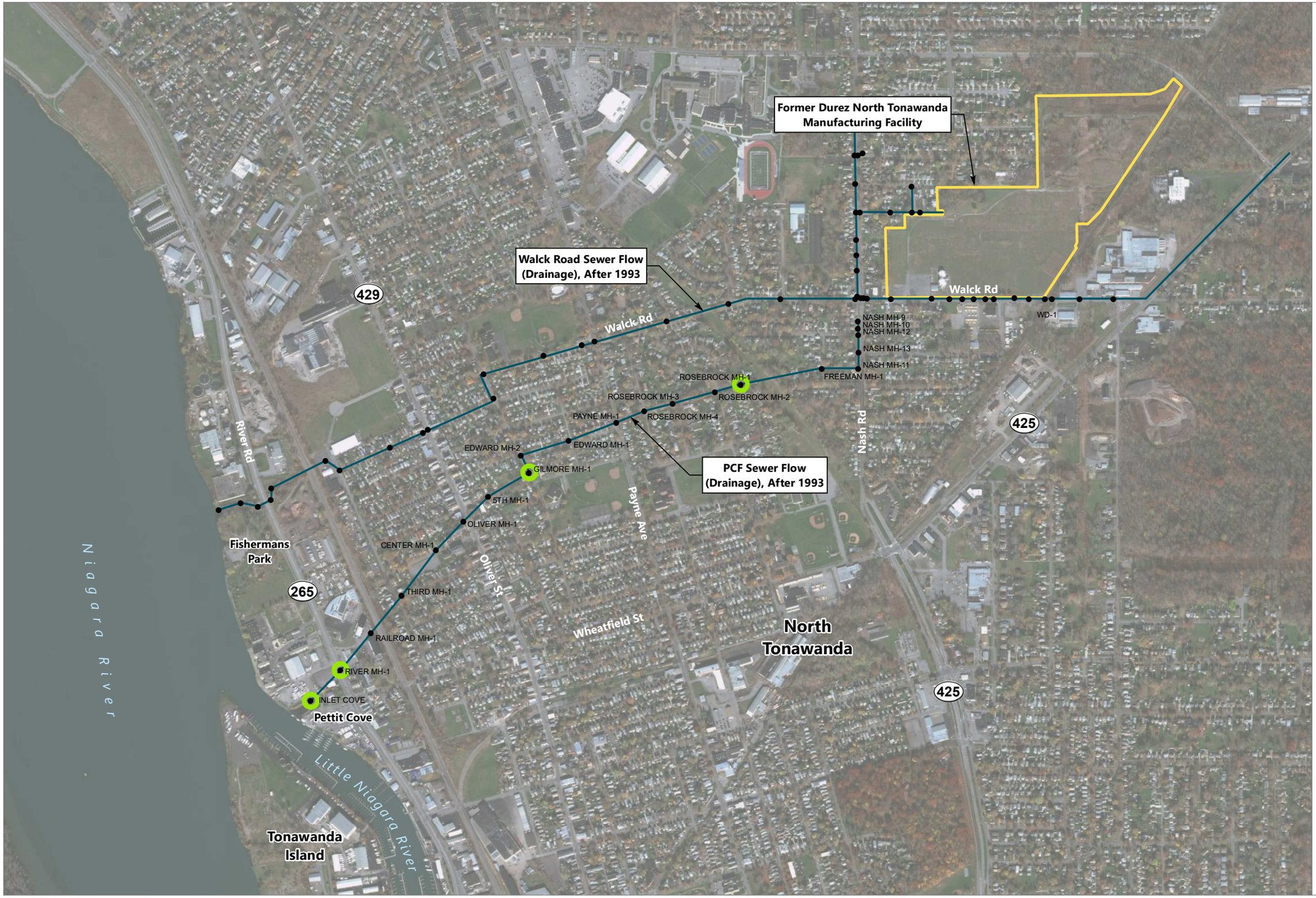
**Figure 3-2**
**Fish and Benthic Community Survey Locations**
Aquatic Assessment Work Plan
Durez Inlet




**LEGEND:**

- Manhole Location
- Target Accumulated Sediment Sampling Location
- Sewer Flow (Drainage), After 1993
- Former Durez North Tonawanda Manufacturing Facility

**NOTE:**
1. Aerial imagery sourced from Esri streaming service, November 2020.

0 2,000
Feet

Publish Date: 2021/07/02, 8:48 AM | User: jquinley
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**Figure 3-3**
**Target Locations for Collection of Accumulated Sediment in PCF**
Aquatic Assessment Work Plan
Durez Inlet

# Appendix A
# Standard Operating Procedure for Ex Situ Porewater Passive Sampling Using LDPE

ANCHOR QEA

## Standard Operating Procedure

# Ex Situ Measurement of Freely Dissolved Concentrations of Hydrophobic Organic Contaminants in Sediment Porewater by Low-Density Polyethylene Sampler

## Scope and Application

This Anchor QEA, LLC, Environmental Geochemistry Laboratory (EGL) Standard Operating Procedure (SOP) is applicable to the ex situ measurement of porewater concentrations of hydrophobic organic contaminants, such as polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), polychlorinated dibenzo-*p*-dioxins and furans (dioxin/furan), and pesticides in sediment porewater with a low-density polyethylene (LDPE) sampler. In this method, the LDPE sampler is placed in the sediment for a sufficient amount of time to allow uptake and equilibration of target analytes in the freely dissolved phase with LDPE. At equilibrium, the dissolved porewater concentrations ($C_w$) can be estimated from the measured concentration absorbed to the LDPE strip ($C_f$) and the LDPE-water partition coefficient ($K_{PE\text{-}w}$), as shown in Equation 1. Performance reference compounds (PRCs) are used to determine the extent to which equilibrium is achieved and, if necessary, to apply a correction for non-equilibrium (Fernandez et al. 2009). Procedures outlined in this SOP will be followed, and any deviations will be noted.

**Equation 1**

$$C_w = \frac{C_{PE}}{K_{PE-w} \times f_e}$$

where:

| | | |
|---|---|---|
| $C_w$ | = | concentration in sediment porewater (nanograms per liter [ng/L]) |
| $C_{PE}$ | = | concentration in LDPE sampler (ng/L) |
| $K_{PE-w}$ | = | LDPE-water partitioning coefficient (liter per liter [L/L]) |
| $f_e$ | = | fraction of equilibrium (-) |

## Health and Safety

All laboratory work will be performed in accordance with the EGL Chemical Hygiene Plan by approved staff. Approval to work in the EGL requires orientation to laboratory safety procedures and potential hazards under the guidance of the Laboratory Manager, as specified in the Chemical Hygiene Plan.

# Equipment and Supplies

The following table provides a list of equipment that may be necessary to carry out the procedures contained in this SOP. Additional equipment may be required.

| Equipment and Supplies | Notes |
|---|---|
| Appropriate personal protective equipment (i.e., safety glasses, disposable gloves, and laboratory coats) | Safety glasses must be used to protect eyes when LDPE sheets are handled. |
| Ponar Grab Sampler | For sediment sample collection |
| Pre-cleaned, stainless steel sampling equipment | Spoons for removing sediment from the ponar grab sampler and bowls for homogenizing the sediment sample |
| Pre-cleaned 32-oz clear glass jar | Approximately 64-ounce of sediment is required for each sample |
| LDPE strip | LDPE sheets (25 micrometers thick) |
| 32-ounce, wide-mouth glass jar with Teflon-lined cap | For PRC spiking |
| Razor blade | To cut LDPE strips into desired sizes |
| Water (high-performance liquid chromatography [HPLC] grade) | Approximately 4 liters (L) is required for washing and PRC spiking. |
| Methanol (HPLC grade) | Approximately 2 L is required for washing and PRC spiking. |
| n-Hexane (HPLC grade) | Approximately 2 L is required for cleaning LDPE strips. |
| Toluene (HPLC grade) | Approximately 2 L is required for cleaning LDPE strips. |
| PRC stock solutions | Contact analytical laboratories in advance to determine whether any PRCs to be used would interfere with analysis of target analytes, surrogates, or internal standards. PRC stock solutions are available from Cambridge Isotope Laboratories, Inc., or Wellington Laboratories Inc.<br>PAHs: deuterated PAHs (fluorene-d10, pyrene-d10, benzo[a]anthracene-d12, dibenz[a,h]anthracene-d14)<br>PCBs: $^{13}$C-labeled PCBs ($^{13}$C-PCB-008, $^{13}$C-PCB-031, $^{13}$C-PCB-060, $^{13}$C-PCB-085, $^{13}$C-PCB-128, $^{13}$C-PCB-182)<br>PCDDs: $^{13}$C-labeled PCDDs ($^{13}$C-1,2,7,8-TCDD, $^{13}$C-1,2,4,7,8-PeCDD, $^{13}$C-1,2,3,4,6,8-HxCDD, $^{13}$C-1,2,3,4,6,7,9-HpCDD) |
| Sodium azide ($NaN_3$) | Added to sediment to prevent microbial degradation of target compounds. This is a hazardous chemical—please carefully review the Safety Data Sheet and handle it in the fume hood. |
| Pre-cleaned, 40-milliliter amber glass volatile organic analysis (VOA) vials | Store LDPE strips to ship to analytical laboratory. |
| 2-milliliter amber glass vials | Store PRC stock solutions. |
| 500-milliliter, wide-mouth glass jars with polytetrafluoroethylene (PTFE)-lined caps | Store sediment samples. |
| Wash bottles | For HPLC-grade water, methanol, n-hexane, and toluene. |
| Laboratory shaker table | Sediment jars are agitated on a shaker table at 60 rotations per minute. |
| Analytical balance | To weigh LDPE strips. |

| Equipment and Supplies | Notes |
|---|---|
| Tweezer | -- |
| Waterproof markers | -- |
| Alconox, Liquinox, or equivalent | -- |
| Micropipette and tips (10 to 1,000 microliters) | -- |
| Heavy-duty aluminum foil | -- |
| Kimwipes | -- |
| Mylar bags | -- |

# Ex Situ LDPE Procedure

## Sediment Sample Collection, Shipping, and Preparation

1. Bulk sediment samples will be collected in the field and shipped to the EGL for ex situ porewater analysis using the following procedure:
    A. Deploy a ponar grab sampler (ponar) though the water column to collect a bulk sediment sample.
    B. Upon retrieving the ponar, measure the recovered sediment sample depth.
        i. If the recovered sediment sample depth is 10 centimeters (cm) or greater, remove the top 10 cm of the sediment sample using a pre-cleaned stainless-steel spoon being sure not to scrape the side walls or the bottom of the ponar to avoid cross-contamination. Sediment removed from the ponar should be placed directly into a pre-cleaned stainless-steel bowl for homogenization.
        ii. If the recovered sediment sample depth is less than 10 cm, use the same methods to remove the sediment sample from the ponar being sure not to scrape the side walls or the bottom and retain the sample.
    C. If the recovered sediment sample depth from step B was less than 10 cm, deploy the ponar again at the same location and measure the recovered sediment sample depth. If the sediment sample depth is less than the sample from step B.ii, discard the ponar sample and retain the sample from step B.ii. If the sediment sample depth is greater than the sample from step B.ii, discard the sample from step B.ii and use the same methods to remove the sediment sample from the ponar and retain it.
    D. If the depth of the sediment sample retained from step C is still less than 10 cm, deploy the ponar one more time at the same location and measure the recovered sediment sample depth. If the sediment sample depth is less than the retained sample from step C, discard the ponar sample and retain the sample from step C. If the sediment sample depth is greater than the sample from step C, discard the sample from step C and use the same methods from Step B to remove the sediment sample from the ponar and retain it.

E. If after three attempts, the recovered sediment sample depth is still less than 10 cm, consult with the project manager about retaining the deepest sample or moving the location.
F. Homogenize the retained sediment sample using a stainless-steel spoon until the sample has a uniform color and texture.
G. Place the sample into two pre-cleaned 32-oz glass jars for LDPE analysis. Sediment should also be placed into an appropriate sample jar for bulk sediment analysis.
H. Collect field duplicate samples at a rate of 1 per 20 samples to assess field precision and LDPE analysis precision. The reproducibility will be determined by calculating relative percent difference as described below.

2. Place sample jars into a cooler and maintain on ice until they can be prepared for shipment to the laboratory.
3. Prior to shipment, refresh the ice to ensure the sample cooler will maintain a temperature of 0°C to 6°C until delivery to the EGL and the chain-of-custody is filled out. A chain-of-custody form will accompany each cooler of samples sent to the EGL.
4. Ship samples to the EGL no later than the day after collection. Samples collected on Friday may be held until the following Monday for shipment.
5. Upon arrival at the EGL, complete the chain-of-custody and process the sediment samples as follows:
   A. Remove coarse particles and debris that might potentially damage LDPE strips from the bulk sediment samples, if necessary. This should be limited to removing non-sorbing constituents like stones, because removal of any sorbing constituents may cause changes in the sediment composition, leading to a matrix that does not fully reflect the in situ conditions.
   B. Add a sufficient amount of the biocide sodium azide ($NaN_3$) to the sediments (producing a concentration of 100 milligrams per liter) to inhibit biological activity during the experiments.
   C. Thoroughly homogenize sediment samples and store in a refrigerator until further analysis. The sediment samples should be stored in the dark to reduce the chance of photodegradation, and stored cold (less than 4°C) to avoid volatilization of target contaminants.

## LDPE Sampler Selection

The LDPE samplers will be created from LDPE sheets (25 micrometers thick). LDPE sheets can be purchased from the following manufacturers: PolyAmerica (Grand Prairie, Texas), Brentwood Plastics, Inc. (Brentwood, Missouri), Carlisle Plastic, Inc. (Minneapolis, Minnesota), Trimaco (Durham, North Carolina), and Film-Gard (Minneapolis, Minnesota) (EPA, SERDP, and ESTCP 2017).

## Detection Limit Calculation

The minimum mass of LDPE needed for specific applications is determined based on the minimum mass of target analytes quantifiable by the chosen analytical method (e.g., gas chromatography [GC]/electron capture detector [ECD] or GC/mass spectrometry [MS] versus high-resolution gas chromatography/high-resolution mass spectrometry [HRGC/HRMS]), the required detection limit for porewater concentrations, and the estimated LDPE-water partition coefficients ($K_{PE-w}$). As shown in Equation 2, the LDPE strip volume ($V_{PE}$) can be converted to mass using the density of LDPE.

**Equation 2**

$$C_{det,PE} = \frac{n_{det}/V_{PE}}{K_{PE-w} \times f_e}$$

where:

| | | |
|---|---|---|
| $C_{det,LDPE}$ | = | method detection limit by LDPE (ng/L) |
| $n_{det}$ | = | minimum mass quantifiable by the analytical method (nanogram [ng]) |
| $V_{PE}$ | = | volume of LDPE strip (liter [L]) |
| $K_{PE-w}$ | = | LDPE-water partitioning coefficient (L/L) |
| $f_e$ | = | fraction of equilibrium (-) |

The introduction of a passive sampler into sediment will inevitably deplete porewater concentrations to some extent. To ensure negligible (less than 1%) depletion of porewater target analytes throughout the course of the experiment and allow for an accurate estimate of porewater concentrations, Ghosh et al. (2014) recommends a mass ratio of 1:100 of polymer to sediment organic carbon (assuming sediment organic carbon and polymer have similar partitioning characteristics).

## LDPE Sampler Preparation

### *LDPE Strip Cleaning and Cutting*

- Determine the mass needed for each LDPE strip in each sample to achieve target detection limits using Equation 2 (see Section 4.3 [Detection Limit Calculation])
- Handling of LDPE strips requires clean nitrile gloves. All work surfaces should be covered with clean, heavy-duty aluminum foil. LDPE processing should be performed in a manner that minimizes background contamination.
- Cut the LDPE strips as needed to fit into a 32-ounce, wide-mouth glass jar for cleaning.
  - Wash a razor blade with water, methanol, n-hexane, and toluene.

- Wash the glass jar with water, methanol, n-hexane, and toluene to remove background contaminants.
- Transfer the LDPE strips into the clean glass jar.
- Wash the LDPE strips with toluene, n-hexane, methanol, and water in the following order:
  - Fill the glass jar with toluene.
  - Agitate for 24 hours in toluene.
  - Discard toluene and fill with n-hexane and agitate for 24 hours.
  - Agitate for 24 hours in n-hexane.
  - Discard n-hexane and fill with methanol and agitate for 24 hours.
  - Discard methanol and fill with reagent water and agitate for 24 hours to remove methanol absorbed by the LDPE strips.
  - Discard water and place the LDPE strips on clean foil and blot dry with Kimwipes.
- Wrap clean LDPE strips with foil and store in a clean, wide-mouth glass jar with Teflon-lined cap.
- Transfer a clean LDPE strip to a pre-weighed volatile organic analysis (VOA) amber glass sample vial for use as a method blank.

## Spiking Performance Reference Compounds

- Place clean LDPE strips in a clean glass jar.
- Prepare a mixture of methanol and reagent water spiked with PRCs. Methanol and water are mixed at a desired ratio (e.g., 80/20 volume per volume [v/v] for dioxin) to optimize PRC spiking (Booij et al. 2002).
- Determine the amount of PRC to spike into the methanol/water solution for each PRC using Equation 3 (Booij et al. 2002). Spike PRCs into the methanol/water solution using a micropipette.
- Add the PRC spiking solution into the glass cylinder and mix well by gentle shaking. Seal the glass jar with minimal headspace.
- Agitate the glass jar to equilibrate for 14 days on a shaker table at 60 rotations per minute.
- After 14 days of agitation, transfer the PRC spiking solution to a different container.
- Fill the glass jar with high-performance liquid chromatography (HPLC)-grade water and leave on a shaker table overnight to remove absorbed methanol from LDPE strips.
- Remove the LDPE strips from the glass jar and blot dry with Kimwipes.
- Cut the LDPE strips to the desired size for deployment if necessary.
- Collect the PRC-loaded passive sampler reproducibility standards in pre-cleaned VOA amber glass vials (n = 3 to 5) and send to an analytical laboratory immediately after recording the masses.

**Equation 3**

$$N_t = nN_m + \frac{N_m V_s}{m_m K_{ms}}$$

where:

| | | |
|---|---|---|
| $N_t$ | = | total amount of PRC to be added to an incubation system (ng) |
| $N_m$ | = | target amount of PRC per LDPE sampler (ng) |
| $V_s$ | = | volume of PRC spiking solution (L) |
| $n$ | = | number of LDPE samplers (-) |
| $m_m$ | = | mass of LDPE sampler (kilogram [kg]) |
| $K_{ms}$ | = | LDPE-methanol/water solution partition coefficient (liters per kilogram [L/kg]) |

## LDPE Sampler Deployment

- Transfer PRC-spiked LDPE strips into the sediment jars with a pre-cleaned metal tweezer.
- Seal the jars with minimum headspace and place into a Mylar bag to minimize photodegradation of target compounds.
- Agitate the sediment jars on a tumbler for a minimum of 30 days.

## LDPE Sampler Retrieval

- Withdraw the LDPE strips from the sediment using pre-cleaned metal tweezers and gloved hand.
- Wash the LDPE strips with HPLC-grade water and wipe with a damp Kimwipe several times to remove any attached sediment particles, biofilms, and mineral deposits.
- Wearing a new set of clean gloves, wipe the LDPE strips again with a dry Kimwipe and transfer to a pre-labeled, pre-weighed amber glass VOA vial.
- Document any color changes on the surface of the LDPE strips. Changes in color may be due to biogeochemical processes in the sediment or the presence of nonaqueous phase liquid (NAPL), which may also be detected by odor. In particular, surface coatings of NAPL can bias results leading to overestimation of freely dissolved concentrations. Note any scratches or other damage to the LDPE strips.
- Record the masses of the LDPE strips using an analytical balance by subtracting the empty VOA vial masses from the masses of the VOA vial containing the LDPE strips. The VOA glass vials are wrapped with bubble wrap, placed in a cooler on ice, and shipped to the analytical laboratory as soon as possible. Samples should be stored at 4°C until chemical analyses are initiated.

# Data Analysis

Porewater concentrations ($C_w$) are calculated from measured LDPE polymer concentrations ($C_{PE}$) according to Equation 1. LDPE-water partition coefficients for target analytes are estimated from a correlation with $K_{ow}$ based on literature $K_{PE\text{-}W}$ and $K_{ow}$ values (Equation 4).

**Equation 4**

$$logK_{PE-W} = a \times logK_{OW} + b$$

where:

$K_{PE-W}$ = LDPE-water partitioning coefficient (L/L)
$K_{OW}$ = octanol-water partitioning coefficient (L/kg)

If equilibrium is not achieved as indicated by the PRC desorption rate, the porewater concentrations are corrected for the fraction of equilibrium ($f_e$) determined from PRC concentrations using the graphical user interface developed by Tcaciuc et al. (2014), which is based on the mass transfer model presented in Fernandez et al. (2009).

# Quality Assurance/Quality Control

## Quality Assurance/Quality Control Samples

Quality assurance/quality control (QA/QC) samples are collected during the ex situ LDPE sampling to ensure contamination is not introduced during the sampling process, including LDPE sampler preparation (pre-deployment), deployment, and retrieval. A summary of the general QA/QC samples is listed below. The number of QA/QC samples per deployment may vary depending on the data quality objectives of the project. In addition, a variety of internal QA/QC checks should be followed in the analytical laboratory.

### *LDPE Method Blank*

An LDPE method blank sample is prepared to assess any residual, analytical background contaminants introduced during cleaning and cutting of LDPE strips. For the method blank, an LDPE strip is cut and cleaned along with other LDPE strips, wrapped with aluminum foil, and stored in an air-tight bag at 4°C until analysis.

### *PRC-Loaded Passive Sampler Reproducibility Standard*

Low variability of PRC concentrations in the PRC-loaded passive sampler reproducibility standards is key for accurately characterizing the fraction of equilibrium of target analytes. PRC-spiked LDPE

samples should exhibit reproducible PRC concentrations. Ghosh et al. (2014) suggest a coefficient of variation less than 20% is acceptable. After spiking PRCs, three to five of the PRC-loaded passive sampler reproducibility standards are immediately sent to SGS North America Inc., in Wilmington, North Carolina, to measure the initial PRC concentrations. The coefficient of variation should be less than 20% (n = 5) at the EGL and reported otherwise.

### *Laboratory Replicate Samples*

Laboratory replicate samples are defined as the following: "Independent samples that are prepared as close as possible to one of the treatability testing samples. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently." They are required as an indication of the reproducibility of the treatability test to be analyzed in the same fashion as the treatability testing samples.

### *Temperature Blank*

Ensure that samples are maintained at the proper temperature during shipping.

## Reproducibility of LDPE Analysis

Laboratory replicate samples will be analyzed at a rate of 1 per 20 samples, or at least one duplicate per analytical batch. The reproducibility will be determined by calculating relative percent difference as shown in Equation 5.

**Equation 5**

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

where:

| | | |
|---|---|---|
| $RPD$ | = | relative percent difference (%) |
| $X_1$ | = | larger result value (ng/L) |
| $X_2$ | = | smaller result value (ng/L) |

## Fouling of LDPE Sampler

Upon retrieval, any color changes in the sampler should be documented. It may be due to changes in sediment biogeochemistry or may indicate the potential that the LDPE strips may have been in contact with NAPL, or have bio-fouling on the surface of the LDPE strips. The use of PRCs may aid in addressing potential artifacts of fouling. If NAPL appears to be present in a sediment sample or on

an LDPE sampler, it should be recorded so that the resulting porewater concentrations will be recognized as potentially affected by artifacts.

## References

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# Appendix B
# Standard Operating Procedure for Fish Community Survey and Tissue Collection

ANCHOR QEA

# Standard Operating Procedure

# Fish Community Survey and Tissue Collection

## Scope and Application

This Standard Operating Procedure (SOP) is applicable to the collection and processing of fish in support of the fish community survey described in the *Aquatic Assessment Work Plan* (Work Plan). The purpose of this SOP is to describe the procedures for fish collection and physical measurements, and preparation of samples for shipment to the laboratory. All fish sample collection described in this SOP is generally consistent with appropriate New York State Department of Environmental Conservation (DEC) guidelines (DEC 2013, 2019a) and handling of fish tissue for chemical analysis is consistent with the procedures described in DEC (2019b).

## Health and Safety Warnings

A safety briefing will be held at the beginning of each day and as new activities are conducted. The designated safety officer shall be responsible for ensuring the safety of personnel and will be contacted immediately in the event of an emergency. Health and safety issues for the work associated with this SOP, including physical and chemical hazards, are addressed in the Health and Safety Plan (HASP). The HASP will be followed during all activities conducted by Anchor QEA, LLC personnel as part of the evaluation.

## Personnel Qualifications

Field personnel executing these procedures will have read, be familiar with, and comply with the requirements of this SOP, the Work Plan, and the HASP. All field personnel are required to take a 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations training course and annual refresher courses and participate in a medical monitoring program prior to engaging in tissue processing activities. Additionally, field personnel will be under the direct supervision of qualified professionals who are experienced in performing the tasks required for sample collection.

## Equipment and Supplies

The following is a list of equipment and supplies that may be necessary for field sampling and in the laboratory, to carry out the procedures contained in this SOP. Additional equipment and supplies may be required, pending field conditions.

### Field Sampling

- Approved documents, including the HASP and SOP
- PPE and clothing as defined in the HASP

- Sampling vessel
- Portable differential global positioning system (DGPS)
- Field laptop or tablet
- Fish community logs
- Ballpoint black ink pens or Sharpie permanent markers (or equivalent)
- Camera
- Sweep nets
- Fiberglass handled dip nets
- Minnow traps
- Anchors for minnow traps
- Hoop or fyke nets
- Backpack electroshocker unit
- Spare Battery
- Beach seine (if needed)
- Waders
- Buckets – for holding fish
- Wader belts
- Waterproof rubberized gloves
- Block nets (if needed)
- Trash Bag – used to dispose of gloves and any other non-hazardous waste generated during sampling
- Plastic (e.g., Gladware) containers
- Fish measuring board or ruler in millimeters
- Portable scale – for weighing fish (capable of accuracy to nearest 0.1 gram)
- Collection tubs or buckets
- Rope
- Latex waterproof, slip-resistant work gloves
- Paper towels
- Duct tape
- Aluminum foil
- Secondary light sources (lamps)
- Stainless-steel utility knife
- Fish identification book
- Coolers and ice
- Sample containers and labels
- Resealable bags
- Nitrile gloves
- Deionized (DI) water

### Laboratory Processing

- Nitrile gloves
- Examination board or tray
- Portable scale (balance) – for weighing fish
- 5% chlorine bleach solution
- DI water

## Sampling Procedures

The following subsections provide step-by-step procedures for the collection and processing of fish.

### Sampler Deployment and Retrieval Procedures

1. Don appropriate personal protective equipment (PPE) as described in the HASP.
2. Determine the appropriate equipment to be used for sample collection requirements prior to field mobilization. Discuss safety issues involved in sampler usage.
3. Perform fish collection with the appropriate equipment. The primary collection methods will be sweep/dip nets, baited minnow traps, hoop nets, as well as backpack electroshocking. If needed, other methods such as beach seines, and angling will be used if necessary.
4. Procedures for the primary collection methods are described in the following:
    - **Sweep/Dip nets**: Sweep nets will be used to collect any fish species. The following protocols will be followed, as practical, for collection of fish with sweep nets:
        - For boat-based sweep net use, position the boat and record the start location using the portable DGPS.
        - Sweep from side to side and from bottom to surface with a controlled steady motion.
        - Bring the net to the surface to check for fish and invertebrates with each sweep.
        - Carefully remove fish from the net as it is pulled onto the boat and place fish into buckets with site water.
        - Process fish as described in the subsequent Sample Processing section.
        - For wading based sweep net use, position the crew along the shore and record the start location using DGPS.
        - Sweep from side to side and from bottom to surface with a controlled steady motion.
        - Bring the net to the surface to check for fish and invertebrates with each sweep.
        - Carefully remove fish from the net and place into buckets with site water.
        - Process fish described in the subsequent Sample Processing section
    - **Baited minnow traps**: Baited minnow traps will be deployed either individually or in a series for collection of forage species. Each deployment will be individually marked by a buoy. Traps will be weighted with appropriate weights. Baited traps will be preferentially deployed during the day or deployed in the late afternoon or evening hours and retrieved

the next morning. The following steps will be taken during deployment and retrieval of minnow traps:

- Position the boat and record the deployment location using the portable DGPS.
- Shore based placement of minnow traps is also acceptable.
- Place the bait into a mesh bag and place the bag in the bait basket of the trap or hook onto the inside center of the trap.
- Attach a buoy to the end of the trap line.
- Lower the trap into the water over the side of the boat or from the shoreline. Ensure the trap is securely anchored on the bottom. The buoy should be clearly visible to allow for ease of retrieval.
- Note the time of deployment, and location of deployment in the field logbook.
- Retrieve traps by hand from the side of the boat or from the shoreline.
- Empty each trap into an individual holding bin or basket.
- Process fish described in the subsequent Sample Processing section.

- **Hoop/fyke nets**: Hoop nets can be deployed either individually or in a series for collection of all fish species. Hoop nets will be positioned parallel to the shoreline. Both hoop nets and fyke nets can be used as passive fish collections methods; however, hoop nets may be better at capturing some of the fish expected to be observed for this project (Flammang et al. 2013). Each deployment will be individually marked by a buoy. Hoop nets will be weighted with appropriate weights. Hoop nets will be preferentially deployed during the day or deployed in the late afternoon or evening hours and retrieved the next morning. The following steps will be taken during deployment and retrieval of hoop nets:
  - Position the boat and record the deployment location using the portable DGPS.
  - Attach a buoy to the end of the net line and ensure the net line is sufficiently long to account for changes in water depth during the deployment period.
  - Lower the net into the water over the side of the boat and place parallel to the shoreline. Ensure the net is securely anchored on the bottom. It is important that the net is stretched taught enough to ensure it stays upright. The buoy should be clearly visible to allow for ease of retrieval.
  - Note the time of deployment, type of net used, and location of deployment in the field logbook.
  - Retrieve nets by hand from the side of the boat.
  - Empty each net into an individual holding bin or basket.
  - Process fish as described in the subsequent Sample Processing section.
- **Boat electroshocking**: Due to the water depths that will exist at some survey areas for this project, the use of a boat-based electroshocker may be needed in some locations. A boat-based electroshocker can be used in nearshore, non-wadable areas. Electroshocking is conducted with a minimum of three individuals. Appropriate PPE will be used by all

individuals involved in electroshocking activities. The PPE includes chest waders and electrical gloves (waterproof rubberized gloves). All individuals involved in electroshocking activities should also have polarized sunglasses. The following steps will be taken during the operation of an electroshocker:

- Record start and end points of the sampling area using the portable DGPS.
- The usual configuration for a boat-based team consists of one individual operating the boat and controlling the shocking unit and two people positioned on the front of the boat to net the fish.
- Only the two individuals positioned at the front of the boat will carry a dipnet.
- The shocked fish may move laterally away, from the electoshocker anode, simply float to the surface, or burrow into the sediment.
- Being able to net the fish with the various movements is how the active method works.
- Place all netted fish into buckets or a live well.
- Process fish as described in the subsequent Sample Processing section.

- **Backpack electroshocking**: Due to the relatively small size of some of the survey areas for this project, the use of a boat-based electroshocker may not be feasible in some locations. Backpack electroshocking (e.g., Smith-Root, LR-24) can be used in nearshore, wadable areas at each location. The backpack unit would not be effective from a boat. Electroshocking is conducted with a minimum of three individuals. Appropriate PPE will be used by all individuals involved in electroshocking activities. The PPE includes chest waders and electrical gloves (waterproof rubberized gloves). All individuals involved in electroshocking activities should also have polarized sunglasses. The following steps will be taken during the operation of an electroshocker:
  - Record start and end points of the sampling area using the portable DGPS.
  - The usual configuration for a small sized team consists of one individual wearing the portable shocking unit, two people positioned on either side of the "shocker" and slightly behind a fourth individual behind the netters to transfer netted fish to a bucket and record data (USEPA 2002). However, the task can be conducted with three individuals. One individual will operate the electroshocker (operator) and two other individuals support the operator. The supporting individuals should usually stay just behind the operator.
  - All three individuals will carry a dipnet. One individual will also carry a bucket.
  - The shocked fish may move laterally away, from the electoshocker anode, simply float to the surface, or burrow into the sediment.
  - Being able net the fish with the various movements is how the active method works.
  - All netted fish will be placed into the bucket.
  - Process fish as described in the subsequent Sample Processing section.

## Field Processing

Fish will be processed on the boat or at the shore. The procedures for processing fish on the boat or at the shore are as follows:

1. Record field information and measurements electronically using the Anchor QEA fish community logs (see Attachment 1). DEC species codes and names are presented in Table 1. During processing, species will be handled as little as possible to promote survival. Once individual processing is complete, live specimens that are not being retained for chemical analysis will be released back into the waterbody.
2. Record the total length of each fish using the measuring board. Place the fish on the board with the anterior end (nose) of the fish against the zero line of the board. Measure the length of the fish at the longest point, from the nose to the end of the compressed tail fin. Record the measurement to the nearest millimeter.
3. Record the weight of the fish. Using a small or large basket for holding the individual (depending on size), tare the balance, and place the fish in the basket. Record the weight of the fish to the nearest 1.0 gram for adult sport fish and nearest 0.1 gram for forage species. Record the length and weight data with the Anchor QEA fish community logs.
4. Composite bluntnose minnow or sunfish by species and by size class (composite samples will be made up of sufficient individuals of similar size, i.e., the smallest individual in a composite is no less than 75% of the total length of the largest individual). Composites will only be made of a single species (not a mix of species).
5. Note any external abnormalities (e.g., deformities, erosions, lesions, tumors, fungus, and parasites) on the fish community logs. Use the U.S. Geological Survey (USGS) guide, Illustrated Field Guide for Assessing External and Internal Anomalies in Fish for reference as needed USGS 2002).
6. Species encountered with questionable identification will be photographed and will either be iced for later examination or identification or preserved in 9% buffered formalin, labeled, and placed in an appropriately sized sample jar for further examination in the laboratory. Formalin will only be used for preservation of fish submitted for taxonomy and will not be used for fish submitted for chemical analysis.
7. Write the fish sample ID, location, date, time, and species on a note card or whiteboard, and photograph the specimen being examined with the sample information. Record this same information with the Anchor QEA fish community logs.
8. Wrap each fish, or multiple fish for composite samples, in clean aluminum foil (shiny side out), and attach an identification label for each individual or composite that includes the sample ID, date, collection method, and location sampled.

9. Place the entire sample into a resealable plastic bag and attach a waterproof identification label on the bag, including the individual sample ID, date, collection method, and sampling zone/area. Place the labeled bag in a cooler on wet ice.
10. Prior to shipping the samples, include a temperature blank in each cooler containing biological samples. Print the chain-of-custody form and include it with the shipment.
11. Print the field collection log and retain a hard copy of the log.
12. Ship samples on wet ice to the analytical laboratory for next day delivery.
13. At the end of the collection event, the laboratory will be instructed to analyze all samples with enough tissue mass for tissue compositing and analysis and archive any that do not meet mass requirements.

## Lab Processing

Laboratory processing of fish tissue samples will follow the procedures described in DEC (2019b) guidance. In summary, forage fish samples will be homogenized upon receipt by the analytical laboratory. Bluntnose minnow or bluegill will arrive at the laboratory in pre-assigned composites, all of which will be prepared in their entirety as a composite sample (e.g., 25 whole-body bluntnose minnows to be processed as a single composite). Details on minimum and preferred sample amount needed, detection limits, list of target compounds is presented in Table 3-1 of the Work Plan. For detection limits see Table 3-4 of the Work Plan.

Sport fish processing of legal-size smallmouth bass (12 inch minimum) or yellow perch (no minimum) will also follow DEC (2019b) guidance for fillet processing, and samples will be bone-in, skin-on, and scales-off, and include both fillets. Filleting will be conducted by an analytical laboratory that is experienced in processing fish for New York State projects. Sport fish will arrive at the laboratory as individuals, that will be individually marked for fillet preparation. Only the left-side fillet (as requested by DEC) will be analyzed if enough tissue mass is available.

## Quality Assurance/Quality Control

Entries in the fish community logs will be double-checked by the field team staff to verify the information is correct. It is the responsibility of the field team leader to periodically check to ensure that procedures are in conformance with those stated in this SOP.

**Table 1**
**Common DEC Species Codes/Names**

| Species Code | Common Name | Species Code | Common Name | Species Code | Common Name |
|---|---|---|---|---|---|
| 0 | No Catch | 390 | Spottail shiner | 576 | White bass |
| 207 | Sea lamprey | 394 | Spotfin Shiner | 576.1 | Temperate Basses |
| 268 | Longnose gar | 396 | Redfin shiner | 591 | Rock bass |
| 271 | Bowfin | 397.1 | Notropis sp. | 595 | Green sunfish |
| 276 | American eel | 400 | Bluntnose minnow | 596 | Pumpkinseed |
| 285 | Blueback Herring | 401 | Fathead minnow | 598 | Bluegill |
| 289 | Alewife | 401.1 | Pimephalus sp. | 599.1 | Lepomis sp. |
| 290.1 | Blueback and/or Alewife | 403 | Longnose dace | 600 | Smallmouth bass |
| 294 | Gizzard shad | 406 | Creek chub | 601 | Largemouth bass |
| 297.1 | Herring Family (Cluepeidae) | 407 | Fallfish | 601.1 | Black Bass (SM or LM) |
| 326 | Rainbow trout | 408.1 | Semotilus sp. | 602 | White crappie |
| 327 | Atlantic salmon | 409.1 | Minnow Family (Cyprinadae) | 603 | Black crappie |
| 328 | Brown trout | 419 | White sucker | 603.1 | Crappie (White or Black) |
| 329 | Brook trout | 423 | Northern hog sucker | 603.2 | Sunfish Family (Centrarchidae) |
| 329.1 | Tiger Trout (hybrid) | 432 | Shorthead redhorse | 613 | Johnny darter |
| 332 | Splake | 433.1 | Suckers (Catostomidae) | 614 | Tesselated darter |
| 332.1 | Trout Family (Salmonidae) | 443 | Yellow bullhead | 616.1 | Ethostoma sp. |
| 335 | Rainbow smelt | 444 | Brown bullhead | 617 | Yellow perch |
| 340 | Central mudminnow | 444.1 | Bullhead (species unknown) | 618 | Logperch |
| 347 | Northern pike | 445 | Channel catfish | 624.1 | Darter (not Y Perch) |
| 349 | Chain pickerel | 450.1 | Freshwater Catfish | 626 | Walleye |
| 350 | Tiger muskellunge | 461 | Trout perch | 628.1 | Perch Family (Percidae) |
| 350.1 | Pike Family (Esocidae) | 493 | Burbot | 700 | Freshwater drum |
| 365 | Carp | 531 | Banded killifish | 970 | NS (Bullhead sunfish, etc) |
| 377 | Golden shiner | 545 | Brook Silverside | 999 | SPECIES UNKNOWN |
| 381 | Emerald shiner | 561 | Brook stickleback | | |
| 385 | Common shiner | 575 | White perch | | |

## References

DEC (New York State Department of Environmental Conservation), 2013. Lake and Pond Fish Community Survey Protocols. NYDEC, Division of Fish, Wildlife and Marine Resources, Bureau of Fisheries. May 14, 2013.

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## List of Attachments

Attachment 1 Fish Community Log

# Attachment 1
# Fish Community Log

# Fish Community Log

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Job:

| | |
|---|---|
| Job No: | Collection Date: |
| Station ID: | Collection Time: |
| Field Staff: | Collection Method: |
| Total # of fish collected at station: | |

| Fish # | Species Code | Common Name | Time | Fish Length (cm) | Whole fish wet weight (blotted) | Notes, including anything on abnormalities |
|---|---|---|---|---|---|---|
| | | | | | | |
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# Appendix C
# Standard Operating Procedure for Benthic Invertebrate Survey and Tissue Collection

ANCHOR QEA

# Standard Operating Procedure

# Benthic Invertebrate Survey and Tissue Collection

## Scope and Application

This Standard Operating Procedure (SOP) is applicable to the collection and processing of benthic community survey samples in support of the benthic community survey and tissue sampling described in the *Aquatic Assessment Work Plan* (Work Plan). All benthic community sample collections will follow appropriate New York State Department of Environmental Conservation (DEC) guidelines (DEC 2019); similarly, the mussel survey will follow the appropriate methods described in DEC (2021), Goforth et al. (2001), and Smith et al. (2001).

## Health and Safety Warnings

A safety briefing will be held at the beginning of each day and as new activities are conducted. The designated safety officer shall be responsible for ensuring the safety of personnel and will be contacted immediately in the event of an emergency. Health and safety issues for the work associated with this SOP, including physical and chemical hazards, are addressed in the Health and Safety Plan (HASP). The HASP will be followed during all activities conducted by Anchor QEA, LLC personnel as part of the evaluation.

## Personnel Qualifications

Field personnel executing these procedures will have read, be familiar with, and comply with the requirements of this SOP, the Work Plan, and the HASP. All field personnel are required to take a 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations training course and annual refresher courses and participate in a medical monitoring program prior to engaging in sample processing activities. Additionally, field personnel will be under the direct supervision of qualified professionals who are experienced in performing the tasks required for sample collection.

## Equipment and Materials

The following is a list of equipment that may be necessary to carry out the procedures contained in this SOP. Additional equipment may be required, pending field conditions.

- Sampling boat equipped with necessary portable navigation, communication, and push core sampling equipment
- Personal protective equipment (PPE) as required by the HASP, including personal floatation devices
- Approved documents, including the SOP and the HASP

- Ballpoint black ink pens or Sharpie permanent markers (or equivalent)
- Digital camera
- Push core sampler
- Aquascope (glass-bottomed bucket)
- Fish measuring board or ruler in millimeters
- Portable scale
- Target sample coordinates
- Water pump and hoses
- Stainless-steel bowls and spoons (or equivalent)
- Appropriate field sieving equipment
- Digital camera
- Surgical forceps
- Ethyl alcohol
- Funnel
- Parafilm
- Whiteboard and marker
- Sample containers
- Sample labels (inside and outside)
- Sample preservation chemicals
- Ruler and tape measure

## Sediment Sample Collection Procedures

As described in the Work Plan, sediment push core samples will be collected from the top 24 inches of the sediment column and segmented into 6-inch increments to support the benthic community survey and tissue sample collection. As described in the Work Plan, it is anticipated that three replicate cores will be needed at each of the proposed locations to obtain sufficient tissue for enumeration and analysis (if needed). Procedures for push core sediment sample collection and processing is provided in Appendix D.

## Collection of the Benthic Community Sample

1. Each 6-inch sediment sample collected for benthic community analysis will be sieved separately.
2. After qualitative characteristics of the sample have been recorded, sediment from the push core segment will be washed on a 0.5-millimeter sieve.
3. Sediment adhering to the outside of the push core should not be mixed with the sample.
4. When being sieved, sediments may be gently sprayed with water from above, gently agitated by hand in a washtub of water (in an up-and-down, not swirling, motion), or washed using a combination of these techniques.
5. It is imperative that the samples be washed gently to minimize specimen damage.

6. If rocks, trash, or larger non-organic pieces of debris are caught in the sieve, rinse thoroughly over the sieve, and carefully remove from the sample.
7. Once sieving is completed, the screen should be held at an angle and the remaining material gently washed into the corner.
8. The sample (i.e., all material on the screen) may then be transferred to a container for immediate fixation, using as little water as possible.
9. Be sure to check the screen for organisms trapped in the mesh wires. Do this by carefully using forceps if needed, taking care not to damage the screen.
10. Place a permanent internal sample label in the container at this time. After the screen has been checked, backwash the screen with a high-pressure spray to dislodge any sediment grains that may be caught in the mesh.
11. To prevent the possibility of breakage, plastic jars and plastic lids will be used to store and ship samples.
12. An identical sample label will be placed on the external surface of the sample jar at this time.
13. Once the entire sample has been sieved and collected in the sample jar, preserve with 98 percent solution of ethyl alcohol (described below).
14. Fill the jar slightly below the threads to ensure all specimens are submerged. After fixative has been added to a sample container, it is critical that the contents be mixed adequately by inverting the container several times.
15. After being stored for approximately 1 hour, samples should be inverted several times again to ensure adequate mixing.

## Benthic Community Fixative Preparation and Identification

1. The fixative appropriate for benthic macroinvertebrate samples is ethyl alcohol.
2. Ethyl or isopropyl alcohol (i.e., preservatives) will be used in place of formalin.
3. A 98 percent solution of ethyl alcohol will be used as the fixative.
4. It will be important to thoroughly sieve the samples before ethyl alcohol addition.
5. Ship samples to benthic laboratory for identification and enumeration.

## Mussel Survey

The following section provides step-by-step procedures for a mussel survey and tissue sample collection. Shoreline searches can be used to survey for mussels in a wadable condition. Visual mussel surveys are conducted along a series of defined transects along the shore (Goforth et al. 2001; Smith et al. 2001; DEC 2021). Shoreline searches using, visual and tactile methods, an aquascope (glass-bottomed bucket), and timed-transects are all common qualitative mussel survey techniques (Goforth et al. 2001; DEC 2021).

1. Wear appropriate PPE as described in the HASP.

2. Determine the appropriate equipment to be used for the survey requirements prior to field mobilization. Discuss safety issues involved in sampler usage.
3. Shoreline searches using an aquascope (glass-bottomed bucket) and timed-transects with visual and tactile survey methods are an appropriate qualitative mussel survey technique (Goforth et al. 2001; DEC 2021).
4. Visual and tactile mussel surveys will be conducted along four defined transects along the shoreline of each mussel survey area defined in the Work Plan.
5. Record the station location coordinates and the water depth on the appropriate data collection form.
6. With respect to level of effort, 15 minutes along a 50 meter transect (1/2 hour for 100 meters) is an appropriate effort.
7. The focus will be on appropriate habitats in a reach while time is being recorded. The exact species of mussels that will be found is unknown. Therefore, several approaches are needed to survey for mussels.
8. The following visual and tactile methods will be used:
    a. Sweeping away silt, sand, and/or small detritus by hand.
    b. Hand-probing at least the upper 5 centimeters (2 inches) of loose substrate. Depending on the potential species within the vicinity of the project site, probing at an additional depth, or searching under rocks may be required.
    c. Searching among the bases/roots of submerged vegetation and around the edges of emergent vegetation.
    d. Searching banks, exposed silt, gravel, or point bars to recover any shells or dead mussels.
    e. Examining any cracks or openings in areas of bedrock, concrete, rip rap, etc.
    f. Thoroughly examining heterogenous substrate (mixtures of sand, cobble, boulders, etc.,)
9. Aquascopes may also be used for underwater viewing within 1 meter of defined transects while wading.
10. Mussels (and dead shells) observed during the timed-transect period are placed in mesh bags, identified to species, measured, enumerated, and released in the field if not retained for tissue analysis.
11. Mussels with open or damaged shells or a siphon tube that does not respond to touch will be considered dead.
12. Measurements of live mussels will include the weight and length of each live mussel.
13. When live mussels are retained for tissue analysis, composite groups of whole mussels will be selected from individuals that are in the mesh bags. The number needed for a composite will vary depending on the species that are present at this site and able to be obtained.

    a. Write the mussel composite sample ID, location, date, time, number of individuals, and species on a note card or whiteboard, and photograph the composite being examined

with the sample information. Record this same information with the Anchor QEA fish community logs.

b. Mussels from each composite sample, is placed in a resealable plastic bag, and labeled appropriately.
c. Composite samples should be kept cool (below 4 degrees Celsius) and shipped overnight to the analytical laboratory within 24 hours after retrieval.
d. Prior to shipping the samples, include a temperature blank in each cooler containing biological samples. Print the chain-of-custody form and include it with the shipment.
e. Print the field collection log and retain a hard copy of the log.
f. Each mussel composite sample will be shucked, weighed, and homogenized at the laboratory.

## Quality Assurance/Quality Control

Entries in the field forms will be double-checked by the field team staff to verify the information is correct. It is the responsibility of the Field Team Leader to periodically check to ensure procedures are in conformance with those stated in this SOP.

## References

DEC (New York State Department of Environmental Conservation), 2019. New York State Department of Environmental Conservation Division of Water Standard Operation Procedure: Biological Monitoring of Surface Waters in New York State. March 2019.

DEC, 2021. New York State Freshwater Mussel Survey Guidelines for Waterbody Disturbance Projects. Prepared by New York State Department of Environmental Conservation. April 2021

Goforth, R.R., D.M. Stagliano, J.G. Cohen, M.R. Pen-skar, Y. Lee, and J.L. Cooper, 2001. *Biodiversity Analysis of Selected Riparian Ecosystems within a Fragmented Landscape*. Report for Michigan Great Lakes Protection Fund and Michigan Dept. of Environmental Quality, Office of the Great Lakes. Michigan Natural Features Inventory report number 2001-06.

Smith, D.R., R.F. Villella, and D.P Lemarié, 2001. "Survey Protocol for Assessment of Endangered Freshwater Mussels in the Allegheny River, Pennsylvania." *Journal of the North American Benthological Society* Vol. 20:118–132.

# Appendix D
# Standard Operating Procedure for Sediment Core Sample Collection and Processing

ANCHOR QEA

# Standard Operating Procedure

# Sediment Core Collection and Field Processing

## Scope and Application

This Standard Operating Procedure (SOP) is applicable to the collection and field processing of high-resolution (i.e., finely-segmented) sediment cores described in the *Aquatic Assessment Work Plan* (Work Plan). This SOP includes procedures for collection of sediment cores using push or gravity coring devices and processing of samples using a core extruding device.

## Health and Safety Warnings

A safety briefing will be held at the beginning of each day and as new activities are conducted. The designated safety officer shall be responsible for ensuring the safety of personnel and will be contacted immediately in the event of an emergency. Health and safety issues for the work associated with this SOP, including physical and chemical hazards, are addressed in the Health and Safety Plan (HASP). The HASP will be followed during all activities conducted by Anchor QEA, LLC personnel as part of the evaluation.

## Personnel Qualifications

Field personnel executing these procedures will have read, be familiar with, and comply with the requirements of this SOP, the Work Plan, and the HASP. All field personnel are required to take a 40-hour Occupational Safety and Health Administration Hazardous Waste Operations and Emergency Response training course and annual refresher courses, as well as participate in a medical monitoring program prior to engaging in sample collection and processing activities. Additionally, field personnel will be under the direct supervision of qualified professionals who are experienced in performing the tasks required for sample collection and processing.

## Summary of Methods

Sediment core collection will be conducted by wading if water depths are shallow, or from an Anchor QEA vessel. In shallow water, cores will be collected by advancing a polycarbonate tube into the sediment with a push coring device. If a vessel is needed because of deeper water, a push core or gravity core device may be used. Exact sampling methods will be determined by conditions encountered in the field. All working surfaces and instruments will be thoroughly cleaned to minimize the potential for cross-contamination between sampling locations. Disposable gloves will be discarded after collecting and processing each sample and replaced prior to handling decontaminated instruments or work surfaces.

## Equipment and Supplies

The following is a list of equipment that may be necessary to carry out the procedures contained in this SOP (additional equipment may be required, pending field conditions):

- Sampling vessel
- Push core or gravity core device
- Differential global positioning system (DGPS)
- 3-inch diameter polycarbonate core tubes
- Caps for core tubing
- Duct tape
- Permanent markers
- Core extruder
- Tape measure
- Taping knives
- Personal protective equipment (PPE) for field team (e.g., rain gear, steel-toed boots, nitrile gloves)
- HASP
- First aid kit
- Cell phone
- Logbooks, indelible black-ink pens
- Resealable plastic bags
- Aluminum foil
- Sample labels
- Chain of custody
- Supplies for sample shipping (e.g., cooler, wet ice, temperature blank, packing tape, custody seals)

## Procedures

The steps for the collection, processing, shipping, and handling of sediment core samples are as follows:

### Sediment Core Collection

1. Navigate to the target sample location with a DGPS that is pre-loaded with target coordinates.
2. When located at the target location, collect and store the coordinates in the DGPS unit.
3. If a vessel is used, anchor in place.
4. Select the appropriate length polycarbonate core tube and use a coring device to advance the tube into the sediment until refusal is met.

5. Remove the core tube from the sediment and place a plastic cap on the bottom of the tube before it breaks the water surface.
6. If the top of the core tube is above the water surface after advancement, use a capped 1-foot section of tubing to fill the core tube with site water from the sampling location and cap and seal with duct tape.
7. With any top caps removed, drain surface water from the core tube and slice the tube at the sediment/water interface. Place a plastic cap on the top of the sliced core and use duct tape to seal the caps and keep them in place.
8. Label each core tube with a permanent marker to include; location ID, sample time, date, and arrow indicating the top of the core.
9. Maintain cores in an upright position during sampling and transport.
10. Store cores out of direct sunlight on wet ice until field processing can be conducted.

## Sediment Core Processing

Core processing consists of removing the recovered material from the core by extruding the core to access the sediments:

1. Take a photograph of the total core length prior to processing.
2. Record a description of the core sample in the field log, including the following as appropriate:
    - Date and time of sample collection
    - Sample recovery (depth interval)
    - Odors (e.g., hydrogen sulfide or petroleum)
    - Visual stratification, structure, and texture
    - Vegetation and debris
    - Photoionization detector readings
    - Presence of sheen
    - Any other distinguishing characteristics or features

3. Extruding the core
    - Remove the bottom end-cap and place the core on the decontaminated extruding device.
    - When the core is in place, remove the upper cap and begin extruding pre-selected depth intervals (2 centimeters [cm] as described in the Work Plan). This will be achieved using a 2 cm template cut from a clean core tube that will be placed on top of the core sample tube.
    - Extrude sediment from the core tube into the template, and insert a stainless-steel taping knife between the tube and template.
    - The sediment sample will be placed onto aluminum foil, homogenized, and then distributed to the proper sample containers.

4. Sample containers obtained from the analytical laboratory will be labeled with the following information:
    - Project name
    - Location ID
    - Date and time
    - Analysis to be performed
    - Samplers initials

5. Thoroughly clean and decontaminate all working surfaces and tools between sample intervals. Decontamination consists of washing with a detergent followed by rinsing with deionized water.
6. Discard disposable gloves and sampling tools after processing each core, and replace prior to handling decontaminated instruments or work surfaces.
7. Complete chain-of-custody documentation
8. Package sample containers into coolers for shipment to the analytical laboratory, including the following:
    - Bubble wrap
    - Wet ice
    - Temperature blank
    - Plastic bag liner
    - Chain of custody

9. Seal coolers with a custody seal and packing tape, and ship to the analytical laboratory priority overnight to arrive next day.