

**BEFORE THE ILLINOIS POLLUTION CONTROL BOARD**

MARATHON PETROLEUM	)	
COMPANY LP,	)	
	)	
Petitioner,	)	
	)	
v.	)	PCB 18-49
	)	(Thermal Demonstration)
ILLINOIS ENVIRONMENTAL	)	
PROTECTION AGENCY,	)	
	)	
Respondent.	)	

**NOTICE OF FILING**

TO: Don Brown	Carol Webb
Clerk of the Board	Hearing Officer
Illinois Pollution Control Board	Illinois Pollution Control Board
100 W. Randolph Street, Suite 11-500	1021 North Grand Avenue East
Chicago, Illinois 60601	P.O. Box 19274
<b>(VIA ELECTRONIC MAIL)</b>	Springfield, Illinois 62794-9274
	<b>(VIA ELECTRONIC MAIL)</b>

**(SEE PERSONS ON ATTACHED SERVICE LIST)**

PLEASE TAKE NOTICE that I have today filed with the Office of the Clerk of the Illinois Pollution Control Board **MARATHON PETROLEUM COMPANY LP'S SUBMITTAL OF REPORT**, a copy of which is herewith served upon you.

Respectfully submitted,  
MARATHON PETROLEUM COMPANY LP,

Dated: October 5, 2023

By:           /s/ Melissa S. Brown            
One of Its Attorneys

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PROTECTION AGENCY,	)	
	)	
Respondent.	)	

**MARATHON PETROLEUM COMPANY LP'S SUBMITTAL OF REPORT**

MARATHON PETROLEUM COMPANY LP ("Marathon"), by and through its attorneys, hereby submits the study report pursuant to the Illinois Pollution Control Board's ("Board") May 26, 2022 Opinion and Order. In support of this filing, Marathon states as follows:

1. On April 7, 2022, the Board entered an Opinion and Order granting Marathon's requested Alternative Thermal Effluent Limitation with conditions.
2. On May 12, 2022, Marathon filed a motion requesting that the Board modify its April 7, 2022 Opinion and Order to extend the deadline for Marathon to perform and submit the deformities, eroded fins, lesions and tumors (DELTs) study required by the Order.
3. On May 26, 2022, the Board issued an Opinion and Order granting Marathon's request for modification, extending the deadline for Marathon to perform and submit the DELTs study to October 7, 2023.
4. Pursuant to Paragraph 5 of the Board's May 26, 2022 Order, Marathon has conducted the study to determine whether Marathon's thermal discharge is causing an increased

incidence of DELTs in the representative important species, including the Bigeye Chub at Robinson Creek. This study was completed by October 7, 2023, as required by the Order.

5. As required by the Board's May 26, 2022 Opinion and Order, Marathon is required to submit a copy of the DELTs study report to the Board, the Illinois Environmental Protection Agency ("Illinois EPA"), and the Illinois Department of Natural Resources ("IDNR").

6. The DELTs study report is attached hereto as Exhibit 1. Marathon hereby submits the DELTS study report by filing the report in this matter and by serving a copy of the report via email on the Board's Clerk and counsel for Illinois EPA and IDNR.

Respectfully submitted,

MARATHON PETROLEUM COMPANY LP,

By: /s/ Melissa S. Brown  
One of Its Attorneys

Dated: October 5, 2023

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**Assessment of Deformity, Erosion, Lesion, and Tumor  
(DELT) Anomalies Associated with the Thermal  
Discharge at Marathon Petroleum Company's  
Robinson Refinery**

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October 2023  
Version: FINAL REPORT  
EA Project No. 1604001

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# Assessment of Deformity, Erosion, Lesion, and Tumor (DELT) Anomalies Associated with the Thermal Discharge at Marathon Petroleum Company's Robinson Refinery

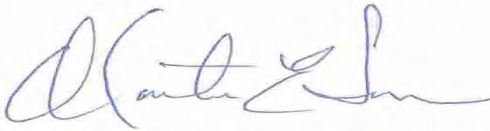
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5 October 2023

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Marty Sneen  
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Date



5 October 2023

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Date

October 2023  
Version: FINAL REPORT  
EA Project No. 1604001

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Appendix C. Water Chemistry Laboratory Report 17 October 2022

**LIST OF ACRONYMS AND ABBREVIATIONS**

AhR	Aryl Hydrocarbon Receptor ligands
ANOVA	Analysis of Variance
ATEL	Alternate Thermal Effluent Limit
°C	Degrees Celsius
CA	California
cat	Catalase
CF	Condition Factor
CON	Bioassay treatment that consisted of exposure to aged and dechlorinated tap water
<i>Cyp1a</i>	Cytochrome P4501A
DELT	Deformity Erosion Lesion Tumor
DNS	Bioassay treatment that consisted of exposure to water from the downstream source at Location 3 in Robinson Creek
DO	Dissolved Oxygen
EA	EA Engineering, Science, and Technology, Inc., PBC
EFF	Bioassay Effluent Location – 100% Robinson Refinery wastewater effluent after treatment and prior to discharge into Robinson Creek (bioassay only)
EF	Electrofishing
FHM	Fathead Minnow
ft	Foot (feet)
g	Gram
gal	Gallon
<i>gst</i>	Glutathione-S-Transferase
IDNR	Illinois Department of Natural Resources
IPCB	Illinois Pollution Control Board
in	Inch(es)
IQR	Interquartile Range
L	Liter
Loc	Location
MA	Massachusetts
MI	Michigan
mg	Milligram
mL	Milliliter
mm	Millimeter
MPC	Marathon Petroleum Corporation
μL	Microliter



**LIST OF ACRONYMS AND ABBREVIATIONS (Cont.)**

$\mu\text{S/cm}$	Microsiemens per centimeter
NCBI	National Center for Biotechnology Information
ng/L	Nanogram(s) per liter
NPDES	National Pollution Discharge Elimination System
NY	New York
OEPA	Ohio Environmental Protection Agency
OK	Oklahoma
PAH	Polyaromatic Hydrocarbon
PCB	Polychlorinated Biphenyls
pg	Picogram
ppm	Parts per Million
QHEI	Qualitative Habitat Evaluation Index
RM	River mile
rpm	Revolutions Per Minute
SN	Seining
<i>sod</i>	Superoxide Dimutase
SOP	Standard Operating Procedure
SS	Spotfin Shiner
SVOC	Semi-Volatile Organic Compounds
UPS	Bioassay treatment that consisted of exposure to water from the upstream source at Location 1 in Robinson Creek
USA	United States of America
USACE	U.S. Army Corp of Engineers
USEPA	U.S. Environmental Protection Agency
VT	Vermont
YOY	Young of the Year

## EXECUTIVE SUMMARY

In response to comments provided by the Illinois Department of Natural Resources (IDNR), the Illinois Pollution Control Board (IPCB) found that the record did not contain adequate information to determine if the synergistic effect of Marathon Petroleum Company's (MPC) Robinson Refinery thermal discharge and non-thermal stressors in Robinson Creek are causing an increased incidence of deformity, erosion, lesion, and tumor (DELT) anomalies on fish. In addition, the presence of the State-threatened Bigeye Chub (*Hybopsis amblops*) in the vicinity of MPC's discharge added to concerns related to the thermal discharge. *Therefore, the primary objective of this study was to determine whether the Robinson Refinery thermal discharge is causing an increased incidence of DELTs on fish in Robinson Creek, particularly the State-threatened Bigeye Chub (Hybopsis amblops).* This study consisted of three elements: 1) an onsite thermal bioassay with Fathead Minnows (*Pimephales promelas*, FHM); 2) fish population and community assessments in Robinson Creek; and 3) health assessment of bioassay Fathead Minnows and Spotfin Shiners (*Cyprinella spiloptera*, SS) collected from Robinson Creek in conjunction with water chemistry analysis of all bioassay test water sources.

For the Fathead Minnow bioassay, fish were chronically exposed to either MPC's 100 percent effluent (EFF), creek water upstream (UPS) or downstream (DNS), plus a control (CON) treatment group of fish kept in aged and dechlorinated tap water. Exposures were conducted in two separate temperature-controlled trailers set either to 20°C or 30°C ( $\pm 2^\circ\text{C}$ ). Water chemistry samples and randomly selected test specimens were collected on Days 30 and 60.

The fish community assessment of Robinson Creek was conducted by electrofishing and seining at three locations: Location 1 upstream of the MPC Outfall 001, Location 2 immediately downstream of MPC Outfall 001, and Location 3 located approximately four miles downstream of MPC Outfall 001. The surveys were conducted in September and October to coincide with Day 30 and Day 60 collections associated with the bioassay and fish health assessments. In addition, aquatic and riparian habitats were evaluated at each location along with physicochemical measures.

The fish health assessment combined both bioassay and field elements. Bioassay Fathead Minnow test specimens and field collected Spotfin Shiners were examined for the presence of DELTs and several endpoints measured at different levels of biological organization to evaluate overall fish health. These measures included body weights and lengths for calculation of condition factor; cortisol levels to evaluate stress; white blood cell differential counts to quantify immune response; lipid content in livers to gauge nutritional condition; and expression of genes related to oxidative stress and chemical exposure. On Day 0, bioassay Fathead Minnow test specimens were randomly selected prior to assignment to CON, UPS, EFF, and DNS. Similarly, Fathead Minnows were randomly collected from the CON, UPS, EFF, and DNS treatments in both the 20°C and 30°C trailers on Day 30 and Day 60. For the field study, Spotfin Shiners were collected by seining from Location 1, Location 2, and Location 3 on Days 30 (i.e., September) and 60 (i.e., October) of the bioassay.

In the Fathead Minnow bioassay, exposure to the 30°C treatments as well as EFF water did not lead to the development of DELT anomalies. In addition, while lower Fathead Minnow survival

was observed among some of the 30°C treatments, this was statistically significant only for the UPS treatments on both Day 30 and Day 60. Water chemistry analyses from each of the test water sources collected on Day 30 and Day 60 were non-detect or similar to background for the analytes examined.

The Robinson Creek fish community assessment yielded 30 species representing nine families combined. The community was dominated by a combination of six minnow and sunfish species, including Silverjaw Minnow, Bluegill, Central Stoneroller, Creek Chub, Bluntnose Minnow, and Green Sunfish. A substantial portion of the catch consisted of highly perturbation tolerant species at all three sampling locations. Collectively, species richness was greater and more variable in October (i.e., Day 60) compared to September (i.e., Day 30), but was consistently higher at the downstream, far-field location (Location 3) and similarly lower at Locations 1 and 2. Bigeye Chub was collected at the furthest downstream location (Location 3) in October by electrofishing (two fish) and seining (four fish). The incidence of DELT anomalies was similarly low at both Location 1 and Location 3 and the Bigeye Chub collected at Location 3 in October did not exhibit DELTs. However, the incidence of DELTs was noticeably elevated at Location 2, immediately downstream of the MPC 001 discharge. Out of 13 species, three exhibited the majority of DELTs at Location 2, and while the vast majority of the DELTs observed at Location 2 were erosion, the incidences of nearly 13% and 21% were notable. In addition to the higher species richness at Location 3, the incidence and severity of DELTs at Location 2 were the most notable spatial differences in the fish community. In terms of habitat, quality was similar at Locations 1 and 3 and notably less at Location 2 due to a lack of instream cover and lower quality riffle development. However, this did not appear to affect the fish community structure since Locations 1 and 2 were similar. Lastly, aside from elevated temperature observed at Location 2 in October, no discernable pattern beyond diel differences was observed among physicochemical measurements.

Among the fish health assessment measures, results varied. Despite there being no evidence that the fish lost appetite or changed their feeding behavior during the study, Fathead Minnow test specimens showed lower growth collectively in the 30°C treatments compared to the 20°C treatments. While Fathead Minnows exposed to the EFF water exhibited increased cortisol and expression of *cyp1a*, these responses were not consistent between Day 30 and Day 60. Across all treatments and both temperatures, there was no evidence of oxidative stress, changes in liver lipids, or notable differences in white blood cell counts. The lack of changes in liver lipids over time for fish held at high temperatures may have contributed to the lower body growth for fish held in the 30°C treatments. An increase in *cyp1a* expression suggests exposure to Aryl Hydrocarbon Receptor (AhR) ligands. However, *cyp1a* induction was also observed in fish exposed to DNS waters and the water chemistry results showed no corresponding increase among signature analytes.

As with the bioassay fish health assessment results, field fish health assessment results varied among measures and over time. However, one measure of consistency was among DELTs. Of the nearly 160 field collected Spotfin Shiners from Day 30 and Day 60 combined, none exhibited DELTs. Although body weights were lower in Spotfin Shiners sampled from Location 2 (downstream of the MPC 001 discharge), this difference was only observed during September.

Spotfin Shiners sampled from Location 2 had increased hepatic lipids and increased *gst* expression but only during October. There was no evidence of increased stress in fish sampled from Location 2 and inconsistent evidence for increased detoxification. Likewise, differential white blood cell counts were normal. Overall, Spotfin Shiners collected from Location 2 were in good health when compared against the Location 1 (upstream of MPC Outfall 001) fish.

Overall, the following observations were made among the three study elements:

- Differences in temperature and treatment did not result in the development of DELTs on Fathead Minnow test specimens during the bioassay.
- Survivability was significantly lower in the UPS 30°C treatment but not in the EFF or DNS 30°C treatments.
- Bioassay treatment water sources were non-detect or similar to background for the water chemistry analytes examined.
- The fish community structure was of similar quality at Locations 1 (i.e., upstream of MPC Outfall 001) and Location 2 (i.e., immediately downstream of MPC Outfall 001).
- DELTs primarily in the form of fin erosion were notably higher at Location 2 in only three species, particularly in YOY Bluegill.
- Due to potential influential factors besides MPC Outfall 001 that were beyond the scope of the IDNR proposed study and this investigation, the cause of the elevated incidence of DELTs in select species and life stages at Location 2 remains to be determined.
- Fathead Minnow test specimens grew less in the 30°C treatments compared to Fathead Minnow test specimens held in the 20°C treatments.
- Fathead Minnow test specimens held in the 30°C treatments exhibited stress in terms of elevated cortisol and expression of *cyp1a* at Day 30 but not at Day 60, which suggests the stress was transitory and the test specimens acclimated to the higher temperature.
- DELTs were not observed on field collected Spotfin Shiner specimens.
- While some spatial differences in fish health markers for the field collected Spotfin Shiners were measured, differences between Location 1 and Location 2 fish were minimal.

These observations indicate that the Robinson Refinery thermal discharge is likely to result in measurable stress on the fish community near the MPC Outfall 001. However, that stress is equally likely to be transient and does not result in community structural changes relative to the community observed upstream of MPC Outfall 001. Further, any transient stress that was observed did not result in the development of DELTs for two species of Leuciscidae that were included in this study and are closely related to the State-threatened Bigeye Chub; Fathead Minnow and Spotfin Shiner. The cause of the elevated incidence of DELTs in select species and life stages at Location 2 remains to be determined due to potential influential factors besides MPC Outfall 001 that were beyond the scope of the IDNR proposed study and this investigation. However, given that DELTs were absent from the collected Bigeye Chub specimens and the absence of DELTs in closely related species (i.e., Fathead Minnows and Spotfin Shiners), it is unlikely that the thermal discharge from MPC Outfall 001 will cause DELTs on Bigeye Chub that may inhabit portions of Robinson Creek.

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## 1. INTRODUCTION

As set forth in Order and Opinion PBC 18-49 and in response to comments provided by the Illinois Department of Natural Resources (IDNR), the Illinois Pollution Control Board (IPCB) found that the record did not contain adequate information to determine if the synergistic effect of Marathon Petroleum Company's (MPC) Robinson Refinery National Pollution Discharge Elimination System (NPDES) permitted thermal discharge and non-thermal stressors in Robinson Creek are causing an increased incidence of deformity, erosion, lesion, and tumor (DELT) anomalies on fish. In addition, the reported presence of the State-threatened Bigeye Chub (*Hybopsis amblops*) in the vicinity of MPC's discharge added to concerns related to the thermal discharge. Given that the proposed alternative thermal effluent limitations (ATELs) include a mixing zone without a zone of passage, the IPCB required as a condition to the ATELs that MPC conduct a study as suggested by the IDNR (PBC 18-49, 7 July 2020 IDNR Response, Attachment C). MPC contracted the team of EA Engineering, Science, and Technology, Inc., PBC (EA) and Purdue University to develop an approach to investigate the relationship between DELT anomalies and temperature. This study was designed to follow the IDNR recommended study with modifications to accommodate field implementation.

Increased prevalence of DELT anomalies in fish has been associated with exposure to stressors including high temperature and pollutants (Post 1983, OEPA 1987). Deformities are defined as anomalies which can include malformation of the head, spinal vertebrae, fins, barbels, or abdomen, and have a variety of causes including, but not limited to, toxic chemicals, heavy metals, viral and bacterial (e.g., *Mycobacterium*) infections, and parasites (e.g., *Myxobolus cerebralis*) (Post 1983; OEPA 2015). Eroding of fins, gill cover, barbels, or other body parts are the result of chronic disease caused principally by flexibacteria invading the tissue and causing necrosis. Necrosis of the fins may also be caused by gryodactylids, a small trematode parasite. Lesions and ulcers appear as open sores or exposed tissue and can be caused by viral (e.g., *Lymphocystis*) and bacterial (e.g., *Flexibacter columnaris*, *Aeromonas*, *Vibrio*) infections. Tumors result from the loss of carefully regulated cellular proliferative growth in tissue and are generally referred to as neoplasia (Post 1983). In wild fish populations, tumors can be the result of exposure to toxic chemicals. For instance, Baumann et al. (1987) identified polycyclic aromatic hydrocarbons (PAHs) as the cause of hepatic tumors in brown bullhead catfish (*Ameiurus nebulosus*) from the Black River in Ohio. Although viral infections (e.g., *Lymphocystis*) can also cause tumors, parasites (e.g., *Glugea anomala* and *Ceratonova shasta*; Post 1983) may cause tumor-like masses, but these are not counted as tumors.

During the 2016 316(a) studies conducted by Midwest Biodiversity Institute (MBI; 2017), elevated incidence of fin erosion was a commonly observed DELT at their sampling location immediately downstream of the MPC 001 discharge. However, the erosion was most severe and prevalent on young-of-the-year Bluegill (*Lepomis macrochirus*).

Therefore, the primary objective of this study was to determine whether the Robinson Refinery thermal discharge is causing an increased incidence of DELTs on fish in Robinson Creek, particularly in Bigeye Chub and similar species. This study consisted of three elements: 1) an onsite thermal bioassay with Fathead Minnows (*Pimephales promelas*, FHM); 2) fish community and habitat assessments in Robinson Creek; and 3) health assessment of bioassay Fathead

Minnows and Spotfin Shiners (*Cyprinella spiloptera*, SS) collected from Robinson Creek in conjunction with water chemistry analysis of all bioassay test water sources. These included body weights and lengths for calculation of condition factor; cortisol levels as a measure of stress; white blood cell differential counts as a measure of immune response; lipid content in livers as a measure of nutritional condition; and expression of genes related to oxidative stress and chemical exposure.

## 2. METHODS AND MATERIALS

The following sections provide an overview of the methods used in this study. A more detailed treatment of the methods is provided in Appendix A.

### 2.1 ONSITE THERMAL BIOASSAY

The onsite thermal bioassay was conducted to examine the response of fish to elevated temperature and various water sources. The primary focus was to determine if higher temperatures contributed to the formation of DELTs on fish.

#### 2.1.1 General Experimental Setup

The onsite thermal bioassay consisted of exposing 6-month-old sexually mature male Fathead Minnow (Section 2.1.2) to three water treatments (100% treated MPC effluent = EFF; upstream = UPS; and downstream = DNS, **Figure 2**) plus a control = CON for 60 days. Water for each of the three treatments and control was exchanged every other day. Water from each of the locations was collected using a dedicated stainless steel submersible pump and was pumped into a 275-gallon plastic tote and transported to the testing trailers. Water was allowed to acclimate to the test temperature for at least 2 hours prior to being pumped from the tote to the reservoir tank in the testing trailers. Prior to the transfer, the reservoir tanks and accumulated waste from the exposure tanks was pumped out to the facilities holding basin. Test vessels consisted of 50-gallon plastic barrels stocked with 25-30 randomly selected Fathead Minnow and with three replicate tanks per water treatment. Additionally, a 300-gallon reservoir of water was recirculated through the testing chambers to increase the total water volume and decrease the total concentration of waste products (**Figure 3**). The system was setup as a modified flow through system, whereby the solution was recirculated through the tanks at a rate of approximately two volume replacements per day. The flow through system constantly replenished chemicals to the exposure system to minimize chemical loss due to chemical degradation or uptake.

Test vessels were setup in two separate environmentally controlled trailers (**Figure 4**) with the same water treatments but at different temperatures. Physicochemical measurements of the test vessels were conducted daily while water chemistry samples were collected from all treatments and both temperatures on Day 0, Day 30, and Day 60. In one trailer, test vessels were maintained at  $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$  while test vessels in the second trailer were maintained at  $30^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . These temperatures represent a background cold water stream maximum condition and an elevated temperature condition that will mimic summer/fall variations. Both trailers were set at 16-hour light and 8-hour dark photoperiods with room temperatures monitored continuously.

#### 2.1.2 Test Organisms

Approximately six-month old Fathead Minnows were obtained from Aquatic BioSystems (Fort Collins, Colorado). This vendor specifically raises the test organisms for scientific use. This specialization ensures that the highest quality, disease-free certified fish will be used to initiate the testing. The culture facilities undergo annual health inspections to ensure animal quality.



EA has selected a commercially reared species, to limit the potential for exposure stress to confound the testing results. The acquisition, holding and testing of field collected organisms can increase the stress and confound the data interpretation. Additionally, since field-collected organisms are not subjected to the same care as cultured organisms, there is the potential for disease and or mortality that would have to be addressed with a holding period prior to the test initiation. Typically, this period ranges from weeks to months depending on the condition of the organisms and would collectively act to confound the results of the test. It is recommended that when choosing test organisms, one should select a species that is representative of resident organisms, sensitive to site contaminants, relevant to the overall assessment endpoints, and consistent with data quality objectives. The test organisms should serve as surrogates for organisms present on the site (USEPA 2002). Based on this framework, EA selected the related commercially available minnow species.

### 2.1.3 Water Quality and Water Chemistry

Temperature, pH, dissolved oxygen, and conductivity were measured daily from one replicate tank per test treatment using a Thermo Scientific Orion Star multimeter (A329, Waltham, MA, USA). Additionally, water chemistry samples were collected at Days 0, 30, 60 and submitted to Pace Laboratories, Indianapolis for 125 priority pollutant chemical analyses, including polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, semi-volatile organic compounds (SVOCs), and metals (**Table 1**). For each of the three collection periods, eight (8) water samples (4 exposure waters x 2 temperatures) were collected and analyzed for the aforementioned parameters. Samples were collected using appropriate Chain-of-Custody Forms as described in the Study Plan (Appendix A).

### 2.1.4 Biological Observations

Each test day, test organisms were observed to record the number of surviving organisms. Dead organisms were removed when observed. Test organisms were observed for the obvious presence of lesions or other deformities and the data recorded. Test specimens were more thoroughly examined during the Day 30 collections and Day 60 collections and termination of the test. If lesions were present, they would have been swabbed and sent for analysis. However, no lesions were observed among the test specimens during any portion of the study. Statistical analyses were performed on the Day 30 and Day 60 survival data according to USEPA (2002) guidance, using the ToxCalc statistical software package (Version 5.0, Tidepool Scientific Software). The data was evaluated using a t-test or Wilcoxon's Two-Sample Test (depending on normal or non-normal data distribution). The statistical analyses were performed to determine if exposure to the samples resulted in significantly lower survival ( $p < 0.05$ ) as compared to the organisms exposed to the corresponding control or Location 1 treatments.

## 2.2 ROBINSON CREEK FISH COMMUNITY ASSESSMENT AND COLLECTIONS

The Robinson Creek fish community assessment was conducted to evaluate the effect of the MPC Outfall 001 temperature on the structure of the fish community as it may relate to the development of DELT anomalies.

### 2.2.1 Sampling Locations

The fish community, habitat, and water quality were assessed at three locations established along a gradient both upstream and downstream of the MPC's Outfall 001 (**Table 2, Figures 5 and 6**).

Sampling locations were established based on proximity to the MPC Outfall 001 thermal discharge and similarity of available habitat quality at the time of sample collection. Sampling locations were documented via a hand-held Global Position System (GPS).

### 2.2.2 Fish Community Assessment

Fish surveys were conducted 20-22 September and 18-20 October 2023 with the September sampling event conducted during the bioassay study. Sampling was designed to capture those seasons when water temperatures were warmest and stream flow generally lowest, compared to other seasons, and therefore represent worst case conditions.

In order to characterize and compare the incidence of DELTs among the three sampling areas, a standardized 200-meter reach was electrofished at each location the day after the fish health specimens were collected via seining. Electrofishing was conducted using either a pram or long-line method. A Smith-Root 1.5 KVA control box provided pulsed DC output powered by a 2,000-watt generator. One crew member operated an electrified probe while another collected stunned fish and monitored the electrofishing system. Due to the close proximity of Locations 1 and 2, additional measures were taken to prevent fish movement between the two locations. A barrier net (seine) was deployed across the entire width of Robinson Creek immediately upstream of the MPC Outfall 001 thermal discharge prior to sampling and remained in place until sampling at Locations 1 and 2 were completed.

All fish collected were identified to species, counted, and examined for DELT anomalies. This information was recorded on a project-specific fish sampling data sheet. The incidence of DELT anomalies were recorded following procedures outlined by Ohio Environmental Protection Agency (OEPA) (2015a and 2015b). Fish identifications were made using An Atlas of Illinois fishes: 150 Years of Change (Metze et al. 2022), and scientific nomenclature followed Metze et al. (2022) and Van der Laan et al. (2022).

No specimens collected by electrofishing were analyzed for the health and condition bloodwork indices because electrofishing can be stressful to fish.

#### 2.2.2.1 Habitat

Habitat at each of the three locations was evaluated using OEPA's QHEI (Qualitative Habitat Evaluation Index) (Rankin 1989; OEPA 2006) as this was the method used for the 316(a) demonstration (MBI 2017). Methods for calculating the QHEI are described in Rankin (1989) and OEPA (2006) and therefore are not discussed in detail here. Principal components (metrics) that are used to develop the QHEI score are:

- Substrate
- Cover
- Channel Morphology
- Riparian Zone and Bank Erosion
- Pool, Riffle, Run Quality
- Stream Gradient

QHEI scores from hundreds of segments around the State of Ohio have indicated that values greater than 60 are generally conducive to the existence of warmwater faunas, whereas scores less than 45 generally will not support a warmwater assemblage consistent with the OEPA warmwater habitat biological criteria (OEPA 1997).

### 2.2.2.2 Water Quality

*In-situ* water quality measurements of water temperature, dissolved oxygen (DO), specific conductance, and pH were collected at mid-depth at each sampling location. Water clarity was measured at each location using a Secchi disk. All water quality measurements were also recorded on the project-specific fish sampling data sheet.

### 2.2.3 DELT Anomalies

A fish DELTs study was conducted as required per IPCB 18-49 to investigate the potential association between the MPC Robinson Refinery thermal discharge at Outfall 001 and incidence of fish DELTs.

DELT anomalies are the group of anomalies for which a clear relationship has been established between their incidence (percentage) and water quality (OEPA 1987). A high frequency of DELT anomalies is a good indication of a stress caused by sublethal stresses, intermittent stresses, and/or chemically contaminated substrates. The source of these stressors can be related to land use practices, point source discharges, non-point sources, and/or a combination of these inputs that may be difficult to discern. OEPA has found that incidence of DELT anomalies less than two percent would be expected for unimpacted locations while levels greater than three to five percent would be considered elevated. The following is an overview of DELT anomalies and their causes in freshwater fishes:

- Deformities – These anomalies can include malformation of the head, spinal vertebrae, fins, barbels, and abdomen, and have a variety of causes including, but not limited to, toxic chemicals, heavy metals, viral and bacterial (e.g., *Mycobacterium*) infections, and parasites (e.g., *Myxobolus cerebralis*; Post 1983) (OEPA 2015a and 2015b).
- Eroded fin, gill cover, barbel, or other body part – These are the result of chronic disease caused principally by flexibacteria invading the tissue and causing necrosis (Post 1983). Necrosis of the fins may also be caused by gryodactylids, a small trematode parasite (OEPA 2015a and 2015b).
- Lesions and Ulcers – These appear as open sores or exposed tissue and can be caused by viral (e.g., *Lymphocystis*) and bacterial (e.g., *Flexibacter columnaris*, *Aeromonas*, *Vibrio*) infections (OEPA 2015a and 2015b).

- Tumors – These result from the loss of carefully regulated cellular proliferative growth in tissue and are generally referred to as neoplasia (Post 1983). In wild fish populations, tumors can be the result of exposure to toxic chemicals. Baumann et al. (1987) identified polynuclear aromatic hydrocarbons (PAHs) as the cause of hepatic tumors in Brown Bullhead from the Black River (Ohio). Viral infections (e.g., *Lymphocystis*) can also cause tumors. Parasites (e.g., *Glugea anomala* and *Ceratonova shasta*; Post 1983) may cause tumor-like masses, but these are not counted as tumors. Parasite masses can be squeezed and broken between the thumb and forefinger, whereas true tumors are firm and not easily broken (OEPA 2015a and 2015b).

#### 2.2.4 Fish Health Collections

Seining was conducted primarily to collect specimens to be evaluated for the stress and health markers portion of the study. Seining was chosen as the collection method in order to minimize stress. Depending on habitat, either a 30-ft bag seine with 1/8-inch Ace mesh or a 10-ft straight seine with 1/8-inch Ace mesh was used. Seining was conducted for up to 90 minutes at each location, depending on the number of target specimens collected. Target fish species were Creek Chub (*Semotilus atromaculatus*), Silverjaw Minnow (*Ericymba buccata*), Spotfin Shiner, and Bluntnose Minnow (*Pimephales notatus*). Only Spotfin Shiners were retained for stress and health analyses as they were the only target species collected in sufficient numbers throughout the study area.

From each location and sampling event, Spotfin Shiner health and condition were assessed by bleeding and collecting tissue samples from 30 individuals per site and time point as described for the Fathead Minnow (Section 2.3; Appendix A) and the same health parameters quantified. Studies have shown that just the process of netting and handling a fish raises plasma cortisol within minutes (Sadoul and Geffroy 2019). Because some of the responses that were measured (i.e., cortisol and gene expression) can change quickly, fish were kept in 5-gal buckets containing cool oxygenated water and sampled as quickly as possible.

### 2.3 FISH HEALTH ASSESSMENT

The fish health assessment was conducted to evaluate biological responses (e.g., fish stress indicators and condition factors) at different levels of organization, from whole organism to molecular responses (Figure 1) as they relate to temperature and different water sources. Similar approaches were used to examine laboratory (i.e., bioassay) and field fish responses to potential stressors.

#### 2.3.1 General Approach

Randomly selected bioassay test fish were collected for health assessments on Days 0, 30 and 60 of the study. A total of 10 fish were sampled on day 0 for determination of baseline values. For Days 30 and 60, several endpoints related to assessing fish health were quantified in five replicates for each water treatment and temperature exposure (40 samples per sampling period). In addition, as described in Section 2.2.4, Spotfin Shiners were collected by seining from the

same three Robinson Creek locations where the fish community assessment was conducted. Nearly 160 Spotfin Shiners were randomly selected, examined, and sampled.

Fathead Minnows and Spotfin Shiners were bled and necropsied as described in the Study Plan (Appendix A) and different types of tissues collected in five replicates for each water treatment and temperature exposure for the assessment of health. Body weight and total length were used to calculate Fulton's Condition Factor, K ( $CF = 100,000 \cdot \text{body weight} / (\text{total length})^3$ ).

### **2.3.2 Fin Cortisol**

Originally, we were planning on quantifying cortisol in plasma. Because of the inability of bleeding some of the fish, we pivoted to quantify cortisol in fins. Based on previous studies, fish scales and fins are a better measure of chronic stress, compared to plasma (Nejad et al. 2019, Kennedy and Janz 2022). Fins were collected from all fish during necropsies. We collected all fins (i.e., dorsal, caudal, both pectoral and both pelvic) from every fish. Fins were washed twice with 1 mL methanol and once with 1 mL ultrapure water to remove foreign sources of cortisol. Next, fins were cut into small pieces and their mass measured. Methanol was added to each fin sample such that samples contained 0.0001 – 0.002 g fin tissue/mL. Fins were left on a shaker for 24 hours at 100 rpm, then centrifuged for 15 minutes at 4500 rpm and 20°C. Methanol was subsequently transferred to another vial to be dried on a sand bath at 60°C. After all the methanol was dried, an additional 1 mL methanol was used to wash down the sides of the vials and vials were dried again under a fume hood. Cortisol levels were quantified using a cortisol Enzyme-Linked Immunosorbent Assay (ELISA) kit sold by Cayman Chemical (Ann Arbor, MI, USA). Samples were run in triplicate using a plate reader (Synergy HTX, BioTek, Winooski, VT, USA) so that an intra-assay Coefficient of Variation (CV%) could be calculated.

### **2.3.3 Differential White Blood Cell Counts**

Differential white blood cell counts were conducted by counting a total of 100 cells from a blood smear. We prepared two blood smears/fish immediately after bleeding the fish in the field, slides were air-dried, fixed with methanol, and stained using Diff-Quick Stain Kits (VWR, Radnor, PA, US) which uses a modified Giemsa stain. Slides were then examined under a light microscope (Nikon Ni-U model, Melville, NY, USA) at 100X under oil. The population of cells counted were classified into either lymphocytes, granulocytes (i.e., neutrophils, eosinophils & basophils) or monocytes.

### **2.3.4 Liver Lipids**

The quantity of total lipids in liver samples was used to assess nutritional condition. A standard gravimetric method from Bligh and Dyer (1959) was adopted for small liver (biopsy) samples to a minimum of 1 mg. The samples were homogenized in a beadbeater for 5 minutes at 2400 rpm (Biospec Products, Mini-Beadbeater-96, Bartlesville, OK, USA) in 300 µL ultrapure water, then 550 µL of methanol and 250 µL of chloroform were added. After letting the samples sit at 4°C for 15 minutes, 250 µL of ultrapure water and 250 µL of chloroform were added. The samples were centrifuged for 10 minutes at 10,000 rpm, resulting in a biphasic solution, upon which the bottom phase containing the lipids was transferred to a clean, aluminum weighing boat and evaporated in

a fume hood for 16-20 hours at ambient temperature. The tubes were then weighed to determine lipid content.

### 2.3.5 Molecular Markers

The expression of two key genes involved in oxidative stress responses was measured: Superoxide dismutase (*sod*) and catalase (*cat*). In addition, expression of cytochrome P4501A (*cyp1a*) and glutathione-s-transferase (*gst*) were quantified as biomarkers of exposure to a wide range of pollutants. Gene expression was quantified using standard qPCR protocols developed at Purdue University (Allmon et al. 2022; Bushong et al. 2023). Beta-actin was used as the reference gene. We assessed the stability in the expression of this reference gene and found its expression to be stable across treatments (**Figure 7**). A list of primers used is presented in **Table 3**. We used published primers for the fathead minnow (Bertucci et al. 2020) but had to develop new ones for the Spotfin Shiner using sequence information from related species using published approaches (Allmon et al. 2022). Spotfin Shiner sequences have been uploaded to National Center for Biotechnology Information's (NCBI) gene bank and Accession Numbers are listed under **Table 3**.

#### 2.3.5.1 RNA Extraction

Following dissection and bisection of the fish liver, a portion was stored in RNeasy lysis buffer, left at ambient temperature for 48-hr, then stored in  $-20^{\circ}\text{C}$  until processed for RNA extraction. All fish livers were individually extracted to obtain RNA using Qiagen RNeasy mini kit (Germantown, MD, USA) according to manufacturer protocols and recommendations for extraction from liver tissue. Individual samples provided adequate RNA yield and purity, which were measured using a Nanodrop 8000 spectrophotometer ( $260/280 = 2.0 \pm 0.2$ ) prior to storage at  $-80^{\circ}\text{C}$ . After thawing, RNA was aliquoted to be treated for DNA contamination with Dnase 1 immediately prior to cDNA synthesis. To protect against degradation, freeze-thaw cycles on RNA for cDNA were limited  $<$  three, as suggested in Vehniäinen and Vornanen (2019).

#### 2.3.5.2 cDNA Synthesis

The biological replicates constituted a bisected portion of a single Fathead Minnow liver, amounting to  $n=5$  for the control treatment at each temperature ( $20^{\circ}\text{C}$  and  $30^{\circ}\text{C}$ ) for Days 30 and 60 and an  $n = 10$  livers from Spotfin Shiner from each site and time point. We did not include Fathead Minnow Day 0 samples for these analyses as we observed RNA degradation in some of these samples. cDNA synthesis was performed with 500 ng total Dnase-treated RNA starting material and SuperScript III reverse transcriptase (Invitrogen, Carlsbad, CA, USA) per manufacturer's instructions.

Synthesized cDNA was stored undiluted at  $-20^{\circ}\text{C}$  until RT-qPCR analysis and diluted to a working concentration of  $2 \text{ ng}/\mu\text{L}$  the morning of analysis.

### 2.3.5.3 RT-qPCR Approach

RT-qPCR primers target genes of interest (*cat*, *sod*, *cyp1a*, *gst*), and reference gene (*b-actin*) were sourced from literature or developed using published NCBI nucleotide data and Primer3Plus software. Primer pair reaction efficiencies were calculated using serially diluted standard curves performed with fish cDNA to ensure primers were validated across species. Optimum annealing temperatures for each primer pair were determined with thermal gradients on a Bio-Rad T100 Thermal Cycler (**Figure 8**). For full primer sequences, NCBI accession numbers, and standard curve parameters, please see **Table 3**.

RT-qPCR was performed on a QuantStudio 3 Real-Time PCR System with iQ SYBR Green Supermix (Bio-Rad, Hercules, CA, USA). We followed the thermal cycling protocols provided by the manufacturer for this Supermix. The starting quantity of cDNA for qPCR analysis was 4 ng/reaction. To protect integrity and handle samples similarly, samples were organized into 96-well plate maps that were individually thawed and RT-qPCR performed to obtain relative mRNA for all genes (*cat*, *sod*, *cyp1a*, *gst*, *b-actin*) on the same Day.

qPCR analysis of gene expression data: Analysis of gene expression was performed calculating relative mRNA for all experimental samples. After calculating relative mRNA, the data were checked for extreme outliers as defined by the 1.5xInterquartile Range grouping by treatment, temperature (for bioassay), and gene, and excluded prior to rechecking assumptions. Expression of target genes relative to the housekeeping gene b-actin was analyzed using the Pfaffl method (Pfaffl 2001, Equation 1).

$$Relative\ mRNA = \frac{E_{target}^{(\overline{Ct}_{RefG} - \overline{Ct}_{Exps})}}{E_{housekeeping}^{(\overline{Ct}_{RefG} - \overline{Ct}_{Exps})}}$$

**Equation 1** – Formula for calculation of Relative mRNA (i.e., Relative Fold Change). E represents the converted primer efficiency of either the target gene or housekeeping gene used for normalization. E is exponentiated to  $\Delta Ct$ , which is computed as the average Ct of the gene in the reference group minus the average Ct of the gene between the technical duplicates of the experimental sample.

### 2.3.6 Statistical Analyses

Statistical analysis of morphometrics and cortisol was completed using factorial independent analysis of variance (ANOVA) (or non-parametric alternative as appropriate) with post-hoc Dunnett contrasts at a fixed threshold of  $p \leq 0.05$  for statistical significance. Statistical analysis of gene expression was completed similarly, with ANOVA on the natural log of the relative mRNA. Analysis was performed by sampling date for the onsite thermal bioassay and the Robinson Creek field study. For the onsite thermal bioassay, the reference group was the control treatment. However, for the field assessment, Location 1 on Robinson Creek was the reference group for analysis. In the case of gene expression data, to improve visualization of these data, fold changes were scaled to the UPS treatment or Location 1. Scaling was conducted by dividing

the fold change for a given target gene of an individual by the average of the UPS treatment or Location 1 for that target gene. This approach appropriately scales the average fold change for the UPS treatment or Location 1 to 1.0 for each target gene.

Model fit was inspected using residual plots to assess for normality and homogeneity of variance. Model fit for gene expression data was additionally inspected to identify influential data points substantially skewing the model's fitted values, classifying data points with a Cook's distance  $\geq 0.5$  as potentially influential. If detected, these influential data points were excluded prior to re-running the model and re-assessing fit. In addition to residual plots, homogeneity of variance was assessed through a Levene's test.



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### 3. RESULTS

#### 3.1 ONSITE THERMAL BIOASSAY

##### 3.1.1 Effects on Survival and Incidence of DELTs

Results of the onsite thermal bioassay testing can be found in **Table 4**.

After Day 30, survival of Fathead Minnow held at 20°C was 100% for UPS, EFF, and DNS treatments and 99% for controls. After Day 60, survival in the UPS, EFF and DNS treatments was 100, 100 and 99%, respectively, while the control treatment had 97% survival. There were no significant differences in survival between treatments in either sampling time.

In contrast, survival was impacted for most treatments when Fathead Minnow were held at 30°C. After Day 30, survival of Fathead Minnow was 81% for the UPS, 92% for the EFF and 100% for the DNS treatments, while the control treatment survival was 99%. Statistical analyses demonstrated that fish survival in the UPS treatment was significantly lower ( $p < 0.05$ ) than the control treatment, while the remaining treatments were not statistically different. Comparison to the UPS treatment indicated that survival did not differ between treatments. After Day 60 of exposure, survival in the UPS, EFF and DNS treatments was 77, 89 and 87%, respectively, while the control treatment had 99% survival. Statistical analyses demonstrated that fish survival in the UPS treatment was significantly lower ( $p < 0.05$ ) than the control treatment, while the remaining treatments were not statistically different. Comparison to the UPS treatment indicated that none of the treatments were significantly lower.

Evaluation of the fish for DELTs indicated that none were observed in either study, except for an observance on Day 5 of a fin erosion for one fish, in the 30°C control treatment. It was noted that no DELTs were observed during the necropsies of the fish at the Days 30 and 60 evaluations.

##### 3.1.2 Water Quality and Water Chemistry

Summaries of water quality parameters can be found in **Table 5**. Physicochemical measures were maintained within acceptable ranges. In addition, water chemistry laboratory reports are presented as Appendix B (Day 30) and Appendix C (Day 60). Among the analytes examined, the results from both sample periods were either non-detect or were similar to upstream and exhibited no discernable pattern.

#### 3.2 ROBINSON CREEK FISH ASSESSMENT

##### 3.2.1 Fish Community Assessment

In total, 30 species representing nine families were collected by electrofishing and seining combined (**Table 6**). In total, 2,437 fish were collected during the electrofishing surveys. The collection was dominated by six minnow and sunfish species: Silverjaw Minnow (28.9 percent), Bluegill (15.9 percent), Central Stoneroller (*Campostoma anomalum*) (15.6 percent), Creek Chub (11.0 percent), Bluntnose Minnow (4.9 percent), and Green Sunfish (*Lepomis cyanellus*)

(4.8 percent) as indicated in **Table 7**. Creek Chub, Bluntnose Minnow, and Green Sunfish are categorized as highly tolerant by Illinois and Ohio regulatory agencies (OEPA 2015, Smogor 2004). Other highly tolerant species collected include White Sucker (*Catostomus commersoni*) and Yellow Bullhead (*Ameiurus natalis*). Thus, approximately 24 percent of the total electrofishing catch was represented by highly tolerant species. In contrast, Shorthead Redhorse (*Moxostoma macrolepidotum*), Bigeye Chub, Sand Shiner (*Notropis stramineus*), Brook Silverside (*Labidesthes sicculus*), Logperch (*Percina caprodes*), and Longear Sunfish (*Lepomis megalotis*) are considered intolerant or moderately intolerant of pollution and represented only about two percent of the total electrofishing catch (**Table 7**).

The six dominant species were generally consistent trip to trip with the exception of Western Mosquitofish (*Gambusia affinis*), which ranked sixth in abundance in October (4.4 percent) compared to eleventh in September (1.5 percent). Bigeye Chub, a State-threatened species, was collected at Location 3, the furthest downstream location, in October by electrofishing (two fish) and seining (four fish). Seining, which was conducted primarily to collect specimens for stress and health marker evaluations, produced the only specimens of Blackstripe Topminnow (*Fundulus notatus*) and Logperch (**Table 8**).

Collectively, species richness was greater and more variable in October compared to September. For example, 19 species were collected in September and ranged from 12 to 16 species among locations compared to 12 to 27 species collected in October (**Table 9**). Species richness was consistently higher at the downstream far-field location, particularly during October (**Figure 6**). In fact, all species encountered in October were collected at Location 3. Many species, particularly larger river species such as Emerald Shiner (*Notropis atherinoides*), Sand Shiner, Channel Shiner (*Notropis wickliffi*), River Carpsucker (*Carpionodes carpio*), Spotted Sucker (*Minytrema melanops*), and Shorthead Redhorse were collected exclusively at the furthest downstream Location 3. The proximity of this location to larger water i.e., Sugar Creek and ultimately the Wabash River may explain the increased fish diversity observed at Location 3.

### 3.2.1.1 Habitat

Habitat quality was comparable at Locations 1 and 3 and slightly poorer at Location 2 with QHEI scores of 64.75, 60.5, and 57.5 respectively (**Table 10**). Based on narrative ratings (OEPA 2006), habitat quality was Good at Locations 1 and 3 and Fair at Location 2. Differences in narrative ratings were due primarily to slightly better cover, pool/current, and riffle/run scores at Locations 1 and 3 compared to Location 2 (**Table 10**). Specifically, more instream cover, deeper pools, and deeper more stable riffle/run complexes were observed at Locations 1 and 3 compared to Location 2.

### 3.2.1.2 Water Quality

Water temperatures in Robinson Creek ranged from 22.7 to 30.5°C in September and much cooler in October ranging from 8.5 to 20.1°C (**Table 11**). Water temperatures were higher at Location 2-EF and Location 2-SN immediately downstream of Outfall 001 compared to upstream and further downstream locations, particularly during October. As is typical, water temperature varied with time of day, cloud cover, air temperature, and/or amount of canopy.

Dissolved oxygen (DO) ranged from 6.5 to 9.8 ppm in September compared to 7.9 to 13.1 ppm in October with little consistent spatial or temporal trends apparent other than values were generally higher in October as expected (**Table 11**). DO measurements at all locations were well-above the Illinois water quality standards (i.e., 5.0 mg/L).

Specific conductance values varied and were generally higher in October averaging 1356  $\mu\text{S}/\text{cm}$  compared to 1307  $\mu\text{S}/\text{cm}$  in September (**Table 11**). Spatially, on days when measurements were collected at all locations specific conductance was marginally higher at Location 2-SN compared to Location 1-SN values but approached ambient values at the far-field downstream Location 3-SN (**Table 11**). The lowest observed value of 961  $\mu\text{S}/\text{cm}$  occurred at Location 1-SN in September, the highest value of 1767  $\mu\text{S}/\text{cm}$  occurred at Location 2-EF, also in September.

Observed pH values were similar among locations and ranged from 7.8 to 8.4 (**Table 11**).

Water clarity was measured with a Secchi disk at all electrofishing locations. However, clarity was greater than depth at all locations. As such, clarity was consistently adequate to see the entire water column during electrofishing and maximum depth was recorded instead of actual Secchi depth.

### 3.2.2 DELT Assessment

A total of 186 fish (7.6 percent of the electrofishing catch), representing nine taxa (29 percent of the taxa collected by electrofishing), exhibited DELT anomalies within the study area during 2022 (**Table 12**). Overall incidence rates were nearly identical between the September and October surveys at 7.6 and 7.7 percent, respectively. Among the eight species afflicted with DELT anomalies (excluding *Lepomis* hybrid as only one specimen was collected/afflicted), incidence rates of DELT anomalies were highest for Bluegill (39.2 percent), Yellow Bullhead (17.4 percent), and Suckermouth Minnow (10.4 percent) and considerably less  $\leq 2.4$  percent for the remaining five species for trips combined (**Table 12**). Aside from a deformity observed on a single Suckermouth Minnow (*Phenacobius mirabilis*), erosion accounted for all DELT anomalies observed (**Table 13**).

The incidence rates of DELT anomalies were consistently highest at Location 2 immediately below Outfall 001 (**Table 12**). Affliction rates ranged from 12.9 to 20.7 percent at Location 2 between the two trips compared to 0.3 to 1.3 percent at Locations 1 and 3. Affliction rates at Location 2 were highest among Bluegill (72.5 to 96.2 percent) and, to a lesser extent, Yellow Bullhead (50 to 75 percent) and Suckermouth Minnow (8.3 to 27.8 percent). Fin erosion observed on Bluegill in Robinson Creek was generally moderate to severe in most instances and was generally restricted to young-of-the-year (YOY) and/or juvenile specimens. Fin erosion is often the result of chronic disease caused by bacteria and is usually absent in least impacted fish communities. It occurs most frequently in areas where fish are chronically exposed to multiple stressors (OEPA 1987, OEPA 2015a and 2015b)

### 3.3 FISH HEALTH ASSESSMENT

#### 3.3.1 Onsite Thermal Bioassay Fish Health Assessment

##### 3.3.1.1 Effects on Body Weight, Total Length and CF of Fathead Minnow

There were no interactions between water treatment and water temperature for these morphological measurements. Temperature had a significant effect on Fathead Minnow growth (**Figures 9, 10, and 11; Table 14**) with fish held at 20°C being heavier and longer at both Days 30 and 60 ( $p < 0.001$  for weight and length) compared to fish maintained at 30°C. Overall, Fathead Minnows held at 30°C weighed  $3.19 \pm 0.06$  g and measured  $63.6 \pm 0.4$  mm compared to fish held at 20°C ( $3.78 \pm 0.06$  g and  $67.9 \pm 0.4$  mm). This decrease in size with increased temperature became more evident over time (i.e., body weights at Day 60 were on average  $3.63 \pm 0.06$  g compared to  $3.33 \pm 0.06$  g on Day 30). Changes in length with temperature were not as obvious which resulted in an overall increase in the CF of Fathead Minnows maintained at 30°C ( $p < 0.001$ ), but only at Day 30 (**Table 15**).

There was also a main effect of water treatment on weight, regardless of temperature (i.e., both 20°C and 30°C), with a significantly decreased weight in Fathead Minnows exposed to the EFF treatment relative to control treatments at both Day 30 ( $p < 0.02$ ;  $3.10 \pm 0.12$  g vs.  $3.54 \pm 0.11$  g) and Day 60 ( $p < 0.001$ ;  $3.15 \pm 0.12$  g vs.  $3.81 \pm 0.12$  g) (**Table 16**). At Day 60, Fathead Minnows exposed to the EFF treatment also had marginally decreased length relative to control treatments ( $p = 0.06$ ;  $65.3 \pm 0.8$  mm vs.  $67.8 \pm 0.8$  mm). This resulted in the EFF treated fish having a marginally decreased CF relative to control treatments on Day 30 ( $p < 0.05$ ) and a significant decrease on Day 60 ( $p < 0.04$ ). In sum, temperature and effluent appear to have contributed to the observed changes in Fathead Minnow body size.

##### 3.3.1.2 Effects on Fin Cortisol, Differential White Blood Cell Counts and Liver Lipid Content

For fin cortisol, the calculated intra-assay CV was 6.1%. At Day 30, fin cortisol was increased in Fathead Minnows held at 30°C ( $p = 0.02$ ;  $3,683 \pm 498$  pg/g compared to  $1,586 \pm 593$  pg/g at 20°C), but this increase was not apparent on Day 60 later (**Figure 12; Table 17**). In addition, on Day 60, there was weak evidence for an interaction between temperature and water treatment ( $p = 0.08$ ) and a post-hoc Dunnett test indicated that, within the 20°C treatment, fish exposed to EFF had increased cortisol levels ( $6,439 \pm 928$  pg/g) relative to control treatments ( $1,974 \pm 804$  pg/g) ( $p = 0.01$ ) (**Table 18**).

The proportion of the different types of white blood cells (i.e., lymphocytes, monocytes, and granulocytes) did not differ between treatments (**Table 19**). As expected, the great majority of the white blood cells (>95%) were composed of lymphocytes.

There were no main effects observed on hepatic lipid content related to water treatment. However, water temperature did significantly influence liver lipid content and Fathead Minnows held at 20°C had lower liver lipid content relative to those held at 30°C for both Days 30 ( $p < 0.03$ ) and 60 ( $p < 0.006$ ) (**Figure 13 and Table 20**). At Day 30, Fathead Minnow maintained at

20°C had a liver lipid percentage of  $3.23 \pm 0.29$ , compared to  $4.23 \pm 0.32$  in fish at 30°C. At Day 60, Fathead Minnow at 20°C had a liver lipid percentage of  $2.81 \pm 0.45$ , compared to  $4.69 \pm 0.44$  in Fathead Minnow at 30°C. Additionally, at Day 30, the effect of water treatment was marginally significant ( $p < 0.05$ ) and the interaction between temperature and water treatment was significant ( $p < 0.02$ ), but post-hoc Dunnett tests did not reveal any significant pairwise differences (**Table 21**).

### 3.3.1.3 Effects on Hepatic Gene Expression

Overall and regardless of sampling time, there were no main effects of water treatment on *cat*, *cyp1a*, *gst*, or *sod* expression relative to control treatments (**Figures 14 and 15; Table 22**). Models with weak evidence of treatment effects ( $p \leq 0.1$ ) on gene expression (*cyp1a*, *sod*) were further evaluated using post-hoc Dunnett tests (**Table 23**). At Day 30, fish exposed to the EFF and DNS water treatments had significantly increased hepatic *cyp1a* gene expression relative to the control treatments ( $p < 0.001$  and  $p < 0.03$ , for each water treatment respectively). However, for *sod*, there were no significant differences in expression detected across water treatments. As with Day 30, Day 60 models with weak evidence of treatment effects ( $p \leq 0.1$ ) on gene expression (*cat*, *sod*) were further evaluated using post-hoc Dunnett tests (**Table 23**). These analyses indicated that while there were no significant differences in the expression of *sod*, there was weak evidence that fish exposed to the EFF may have lower *cat* gene expression relative to the control treatments ( $p < 0.06$ ).

Independent of water treatment, hepatic *gst* expression at Day 30 for Fathead Minnow exposed to 30°C significantly decreased relative to 20°C ( $p < 0.03$ ) (**Table 22**). Similarly, hepatic *gst* expression at Day 60 for Fathead Minnow exposed to 30°C was significantly lower relative to 20°C, regardless of the water treatment ( $p < 0.002$ ).

## 3.3.2 Robinson Creek Fish Health Assessment

### 3.3.2.1 Prevalence of DELTs on Spotfin Shiner

Out of the 157 Spotfin Shiner examined during necropsies, we found one fish with a nematode (likely *Strongyloides* spp. or “thread worm”) and no DELT anomalies.

### 3.3.2.2 Effects on Body Weight, Total Length and CF of Spotfin Shiner

There was a significant main effect of location on fish weight with Spotfin Shiners sampled from Location 3 having significantly lower body weights during both September ( $p < 0.007$ ;  $1.97 \pm 0.21$  g) and October ( $p < 0.001$ ;  $1.76 \pm 0.24$  g) relative to Location 1 ( $2.96 \pm 0.25$  g for September and  $3.18 \pm 0.32$  g for October) (**Figure 16; Tables 24, 25**). In addition, Spotfin Shiner from Location 2 (i.e., immediately downstream of MPC Outfall 001) had a lower body weight compared to Location 1 Spotfin Shiners, but only during the September sampling ( $p < 0.05$ ;  $2.23 \pm 0.21$  g).

Spotfin Shiners sampled from the Location 3 were shorter during the October sampling ( $p < 0.001$ ;  $62.0 \pm 1.9$  mm vs.  $74.0 \pm 2.5$  mm from Location 1), but no differences were observed in

September (**Figure 17; Tables 24, 25**). During September, there was a significantly decreased CF in Spotfin Shiners from the Location 3 ( $p < 0.04$ ;  $0.70 \pm 0.01$ ) and a marginal increased CF in Spotfin Shiners from the Location 2 ( $p < 0.07$ ;  $0.78 \pm 0.01$ ) compared to Location 1 ( $0.74 \pm 0.01$ ). In October, CF was significantly increased in Spotfin Shiners sampled from the Location 2 ( $p < 0.001$ ;  $0.88 \pm 0.01$ ) relative to Location 1 ( $0.76 \pm 0.01$ ) (**Figure 18**).

### 3.3.2.3 Effects on Fin Cortisol, Differential White Blood Cell Counts, and Liver Lipid Content

For fin cortisol, the calculated intra-assay CV was 5.0%. There were no differences in cortisol levels in Spotfin Shiner in relation to location or time of sampling (**Figure 19; Table 26**). Similarly to what was observed in Fathead Minnows, there were no significant differences in white blood cell differential counts in Spotfin Shiners sampled from Location 2 and Location 3 relative to Location 1 (**Table 18; Table 27**).

From the September sampling, Spotfin Shiners from the Location 3 had a significantly decreased liver lipid percentage relative to the Location 1 ( $p < 0.03$ ;  $1.57 \pm 0.22$  vs.  $2.35 \pm 0.20$ ) (**Figure 20; Tables 28, 29**). Also, Spotfin Shiners sampled from the Location 2 had a significantly increased liver lipid percentage relative to the Location 1, but only during the October sampling ( $p < 0.009$ ;  $2.94 \pm 0.26$  vs.  $1.74 \pm 0.28$ ).

### 3.3.2.4 Effects on Hepatic Gene Expression

Expression of *cat*, *sod*, *cyp1a*, and *gst* did not significantly change relative to the Location 1 in Spotfin Shiners sampled during September (**Figure 21; Table 30**). However, there was evidence for upregulation in *gst* expression in fish collected at the Location 2 in October compared to Location 1 ( $p < 0.03$ ) (**Figure 22; Table 31**).

## 4. DISCUSSION

### 4.1 FISH COMMUNITY ASSESSMENT AND COLLECTIONS

In September, Location 2 had a mean water temperature of 30.2°C compared to 25.1°C at Location 1. In October, the water temperature at Location 2 was noticeably higher (19.9°C) compared to Location 1 (8.7°C). Habitat quality at Location 2 was ranked as fair, primarily due to lower cover, pool/current, and riffle/run scores.

Species richness was highest at Location 3 and increased in the October sampling. Close to 30 percent of the fish taxa sampled (which corresponds to eight species) had DELTs and the overall prevalence was similar between the September (7.6%) and October (7.7%) sampling events. In Ohio, studies have found that three to five percent incidence of DELTs is considered elevated compared to expected levels (OEPA 1987). While a variety of DELT anomalies were observed during the field study, the majority consisted of fin erosion, which was more prevalent in Bluegill collected at Location 2. The overall incidence of fin erosion at Location 2 ranged from 12.9 to 20.7 percent, compared to 0.3 to 1.3 percent in fish from Location 1 and Location 3. Fin erosion was generally observed in YOY Bluegill at Location 2 and ranged from 72.5 to 96.2 percent.

No DELT anomalies were observed during the bioassay or fish health assessment with Fathead Minnows. Similarly, no DELTs were observed on Spotfin Shiners sampled for the fish health assessment from the three study locations.

Elevated incidence of DELT anomalies is typically related to sublethal stressors related to land use practices, point source discharges, and/or non-point sources. The cause of the elevated incidence of DELTs in select species and life stages at Location 2 remains to be determined due to potential influential factors besides MPC Outfall 001 that were beyond the scope of the IDNR proposed study and this investigation.

### 4.2 ONSITE THERMAL BIOASSAY AND FISH HEALTH ASSESSMENT

In the bioassay, survival of Fathead Minnows was similar among all treatments and during both periods for the 20°C test. In contrast, survival was generally lower in the 30°C test but was only significantly lower in the UPS treatment.

The decreased growth in Fathead Minnow held chronically at 30°C was not surprising given that the chronic water temperature threshold for this species is 29°C (NDEP 2016). Because of this trend, a decline in liver lipid content was also expected for the fish held at 30°C. Instead, the opposite was observed, and hepatic lipid content significantly increased in Fathead Minnows held at 30°C (~ 30 percent increase). Interestingly, the lipids in livers of fish held at 20°C decreased from Day 30 to Day 60 (from 3.23 to 2.81 percent) compared to an increase for the 30°C group (from 4.23 to 4.69 percent).

Fathead Minnows held in the EFF treatment, regardless of temperature, grew less and on average weighed less compared to control treatments ( $3.12 \pm 0.09$  g versus  $3.67 \pm 0.08$  g) which



represents a 15 percent decline in body weight. This difference occurred despite the fact that all fish were fed *ad libitum* and no changes in feeding behaviors (e.g., decreased appetite) were observed during the experiment.

Differences in size among the field collected Spotfin Shiners from Robinson Creek appeared to support the bioassay observations as the body weight of Location 2 Spotfin Shiners was 25 percent lower than Location 1 fish. However, this was only observed in October. Spotfin Shiner from Location 3 were smaller compared to Location 1 during both sampling events.

There were no significant effects of water treatment on hepatic lipid levels of Fathead Minnow. However, Fathead Minnow exposed to higher temperatures had higher liver lipid levels. For the field study, liver lipid was higher in Spotfin Shiner sampled from Location 2, but only during the October sampling, whereas Spotfin Shiner sampled from Location 3 has decreased liver lipids during the September sampling.

Cortisol fin data suggests Fathead Minnow experienced transitory stress at Day 30 when held at 30°C but may have been able to compensate for this stress over time due to a lack of response after Day 60. In terms of water treatment, chronic exposure to EFF water may have contributed to elevated cortisol for fish exposed at ambient temperature (20°C), but there is no indication of combined environmental stressors influencing cortisol levels. Due to elevated cortisol level variability among individuals, an increased sample size would likely be necessary to clarify these trends. In addition, the relationship between fish size/age and cortisol levels is not well understood and likely varies by species. For instance, scale cortisol concentrations have been reported to be independent of fish size (d'Orbcastel et al. 2021) but have also been reported to be impacted by fish weight, with heavier fish showing higher basal plasma cortisol concentrations (Alfonso et al. 2023). Therefore, future cortisol analysis with the Spotfin Shiner should use fish size as a covariate.

Among the fish health parameters that exhibited positive results was white blood cells, which may elevate or become disproportionate in response to disease or infections that may result in DELTs. However, the proportions of the different types of white blood cells (i.e., lymphocytes, monocytes, and granulocytes) in Fathead Minnow and Spotfin Shiner did not differ among treatments or location. The majority of the white blood cells was composed of lymphocytes (> 95%). Although no additional immune related endpoints were measured, the lack of mortality and DELTs in the Fathead Minnow bioassay and absence of DELTs among the nearly 160 Spotfin Shiner examined, are consistent with the absence of disease or obvious infection that was observed.

In the bioassay, Fathead Minnow exposed to the EFF and DNS treatments for Day 30 responded with an increase in *cyp1a* expression. There was also a significant effect of temperature on the expression of *gst*. Temperature decreased the expression of *gst* at 30°C relative to 20°C. Induction of *cyp1a* in fish as a biomarker of exposure to aryl hydrocarbon receptor (AhR) ligands (such as PAHs present in crude oil) and pharmaceuticals are well-studied phenomenon and have been applied in numerous lab and field studies (Bucheli and Fent 1995, Burkina et al. 2015, Anleeb et al. 2023). Despite the fact that water chemistry samples showed no differences

among the locations (Appendix B and C), the EFF treatment results are consistent with several studies, including from studies with Fathead Minnow exposed to PAHs (LaPlaca et al. 2020). Notably, *cyp1a* was also induced in the DNS treatment, suggesting the presence of PAHs and/or other AhR organic ligands. The fact that *cyp1a* expression levels were only slightly increased and returned back to control treatment levels by Day 60, implies that fish were likely exposed to low levels of AhR agonists and acclimated to this exposure. While exposure to hydrophobic compounds like PAHs may be through routes other than water, the lack of PAHs in test water from the different water treatments evaluated (i.e., all values reported were non detected) support these findings.

In contrast to what was observed with Fathead Minnows, we observed a significant effect of location for *gst* with an upregulation at Location 2 relative to Location 1. This pattern was not consistent across sampling times as we did not observe this upregulation in the September sampling. Although there is evidence in the literature for directionality of *gst* activity in response to chemical exposure, there are also studies indicating important seasonal variations in the expression of this gene (Tsangaris et al. 2011) potentially raising questions of whether *gst* may be robust enough for use in field studies. Therefore, the lack of corresponding upregulation of *cyp1a* in Spotfin Shiner suggests minimal exposure to AhR organic ligands. These results also suggest that while oxidative stress in the study fish was observed (i.e., *gst*), this stress did not produce a species or community level effect.

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## 5. CONCLUSIONS

### 5.1 FISH COMMUNITY ASSESSMENT AND COLLECTIONS

Location 2 provides fair habitat for fish compared to the good habitat at Locations 1 and 3. Species richness was generally poor but similar at Locations 1 and 2 compared to Location 3. However, tolerant, and highly tolerant species dominated the catch at each of the three locations. Tolerant species are often a major component in small stream fish communities, like Robinson Creek. DELT anomalies were observed at all three locations and during both September and October among eight species and one hybrid sunfish. A variety of DELTs were observed during the surveys and the vast majority was fin erosion on YOY Bluegill at Location 2, during both September and October. While the magnitude of fin erosion in YOY Bluegills at Location 2 is substantial and the cause remains unclear, the corresponding lack of DELTs on the majority of other species and absence of DELTs on the study fish suggests that the State-Threatened Bigeye Chub will be similarly unaffected.

### 5.2 ONSITE THERMAL BIOASSAY AND FISH HEALTH ASSESSMENT

Exposure to EFF treatments did not lead to the development of DELT anomalies. In addition, while lower Fathead Minnow survival was observed among some of the high temperature exposures, this was statistically significant only for the UPS treatments on both Day 30 and Day 60, whereas growth declined for Fathead Minnow exposed to the EFF treatments at both temperatures, relative to the control treatments. In addition, Fathead Minnow exposed to the EFF treatments responded with increased cortisol and expression of *cyp1a*, although these responses were not consistent and varied depending on time of sampling. There was no evidence of oxidative stress or changes in liver lipids and differential white blood cell counts were normal. Overall, Fathead Minnows chronically exposed to the EFF treatments grew less and likely experienced stress. The significantly increased mortality among the UPS higher temperature treatment was not related to an increase in DELT anomalies as none were detected during this study. There was also no evidence that the fish lost appetite or changed their feeding behavior during the study. While a number of biochemical factors influence growth rates, the absence of changes in liver lipids over time for fish held at high temperatures may have contributed to the lack of body growth in these fish as the liver is a major reserve of lipids. The increase in *cyp1a* expression in EFF and DNS Fathead Minnows suggests exposure to AhR ligands. However, water chemistry analyses from each of the test water sources collected on Day 30 and Day 60 were non-detect or similar to background for the analytes examined.

Fish health measures were collected from Robinson Creek Spotfin Shiner since they represent a similar and abundant species found throughout the study area. Of the 157 Spotfin Shiners that were examined, no DELTs were observed. Although body weight decreased in Spotfin Shiners sampled from Location 2, this pattern was only observed during September. Spotfin Shiners sampled from Location 2 also had increased hepatic lipids and increased *gst* expression during October. However, there was no support for increased stress or oxidative stress in fish sampled from Location 2 and minor evidence for increased detoxification as only the expression of *gst* was increased. Differential white blood cell counts were normal. Overall, Spotfin Shiners collected from Location 2 exhibited comparable health to Location 1 fish.

### 5.3 SUMMARY OF FINDINGS

Overall, the following observations were made among the three study elements:

- Differences in temperature and treatment did not result in the development of DELTs on Fathead Minnow test specimens during the bioassay.
- Survivability was significantly lower in the UPS 30°C treatment but not in the EFF or DNS 30°C treatments.
- Bioassay treatment water sources were non-detect or similar to background for the water chemistry analytes examined.
- 
- The fish community structure was of similar quality at Locations 1 (i.e., upstream of MPC Outfall 001) and Location 2 (i.e., immediately downstream of MPC Outfall 001).
- DELTs primarily in the form of fin erosion were notably higher at Location 2 in only three species, particularly in YOY Bluegill.
- Due to potential influential factors besides MPC Outfall 001 that were beyond the scope of the IDNR proposed study and this investigation, the cause of the elevated incidence of DELTs in select species and life stages at Location 2 remains to be determined.
- Fathead Minnow test specimens grew less in the 30°C treatments compared to Fathead Minnow test specimens held in the 20°C treatments.
- Fathead Minnow test specimens held in the 30°C treatments exhibited stress in terms of elevated cortisol and expression of *cyp1a* at Day 30 but not at Day 60, which suggests the stress was transitory and the test specimens acclimated to the higher temperature.
- DELTs were not observed on field collected Spotfin Shiner specimens.
- While some spatial differences in fish health markers for the field collected Spotfin Shiners were measured, differences between Location 1 and Location 2 fish were minimal.

These findings indicate that the Robinson Refinery thermal discharge is likely to result in measurable stress on the fish community near the MPC Outfall 001. However, that stress is equally likely to be transient and did not result in community structural changes relative to the community observed upstream of MPC Outfall 001. Further, any transient stress that was observed did not result in the development of DELTs for two species of Leuciscidae that were included in this study and are closely related to the State-threatened Bigeye Chub; Fathead Minnow and Spotfin Shiner. The cause of the elevated incidence of DELTs in select species and life stages at Location 2 remains to be determined due to potential influential factors besides MPC Outfall 001 that were beyond the scope of the IDNR proposed study and this investigation. However, given that DELTs were absent from the collected Bigeye Chub specimens and the absence of DELTs in closely related species (i.e., Fathead Minnows and Spotfin Shiners), it is unlikely that the thermal discharge from MPC Outfall 001 will cause DELTs on Bigeye Chub that may inhabit portions of Robinson Creek.

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## **Figures**

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**Figure 1.** Six levels of biological organization were included in paired laboratory and field fish studies.

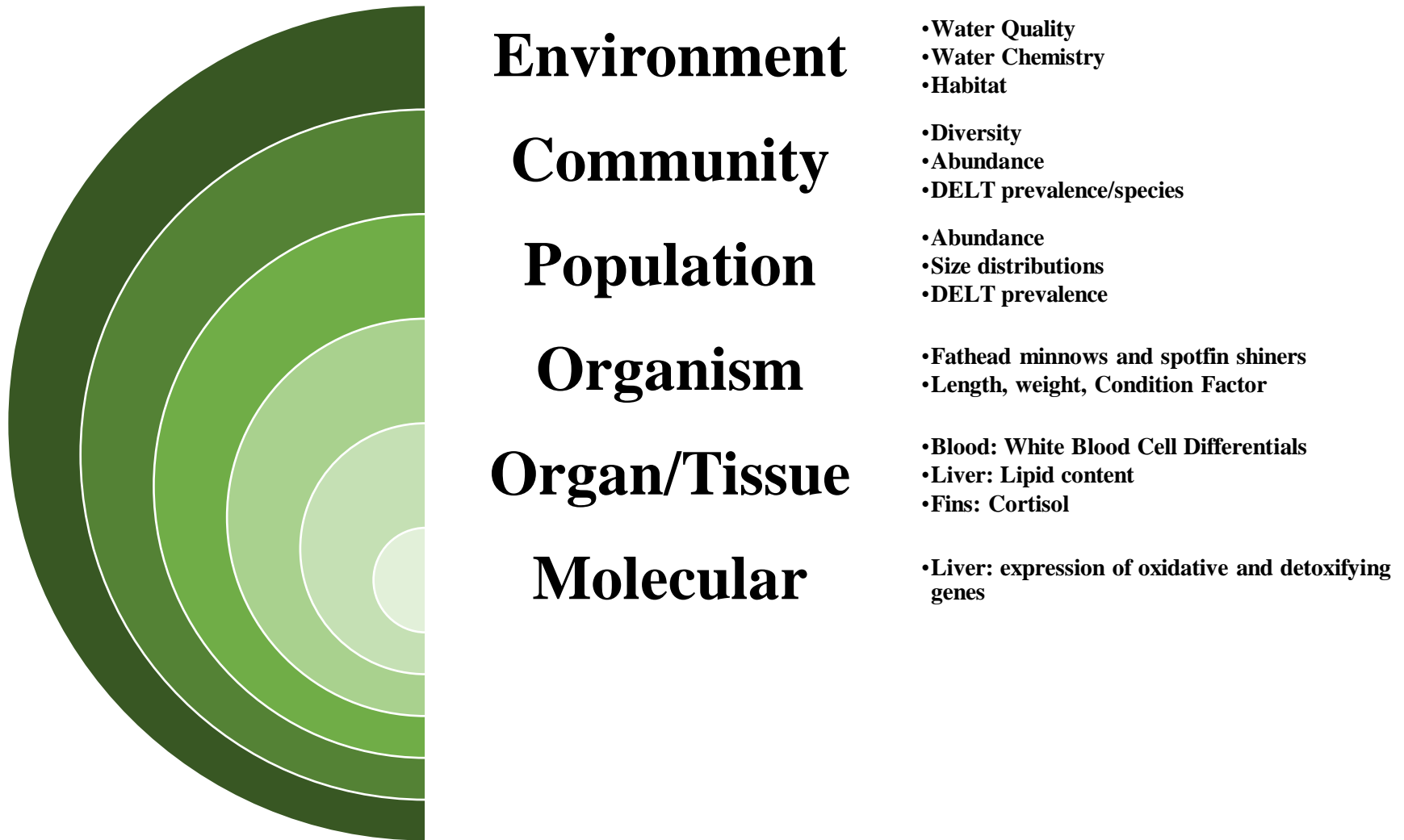
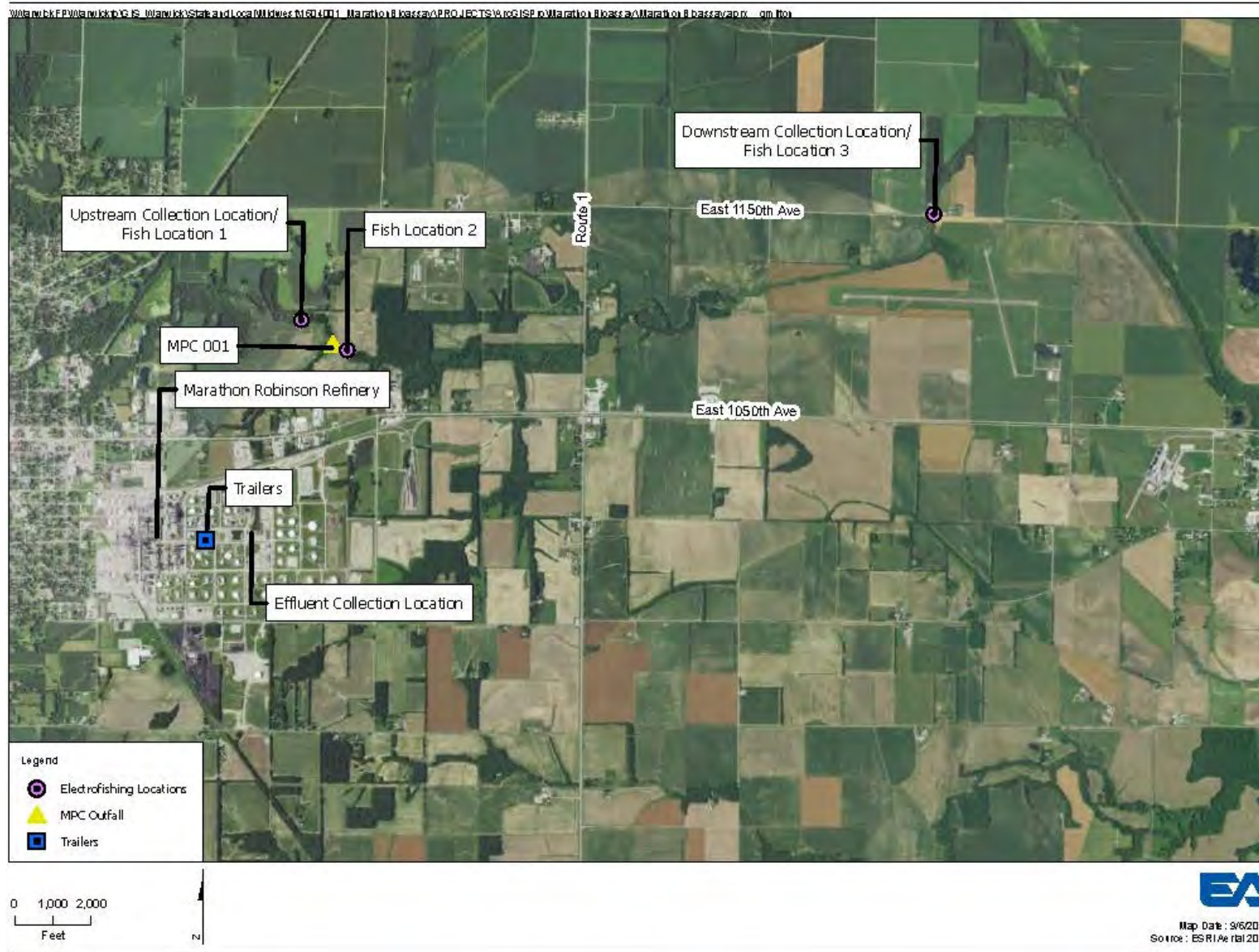
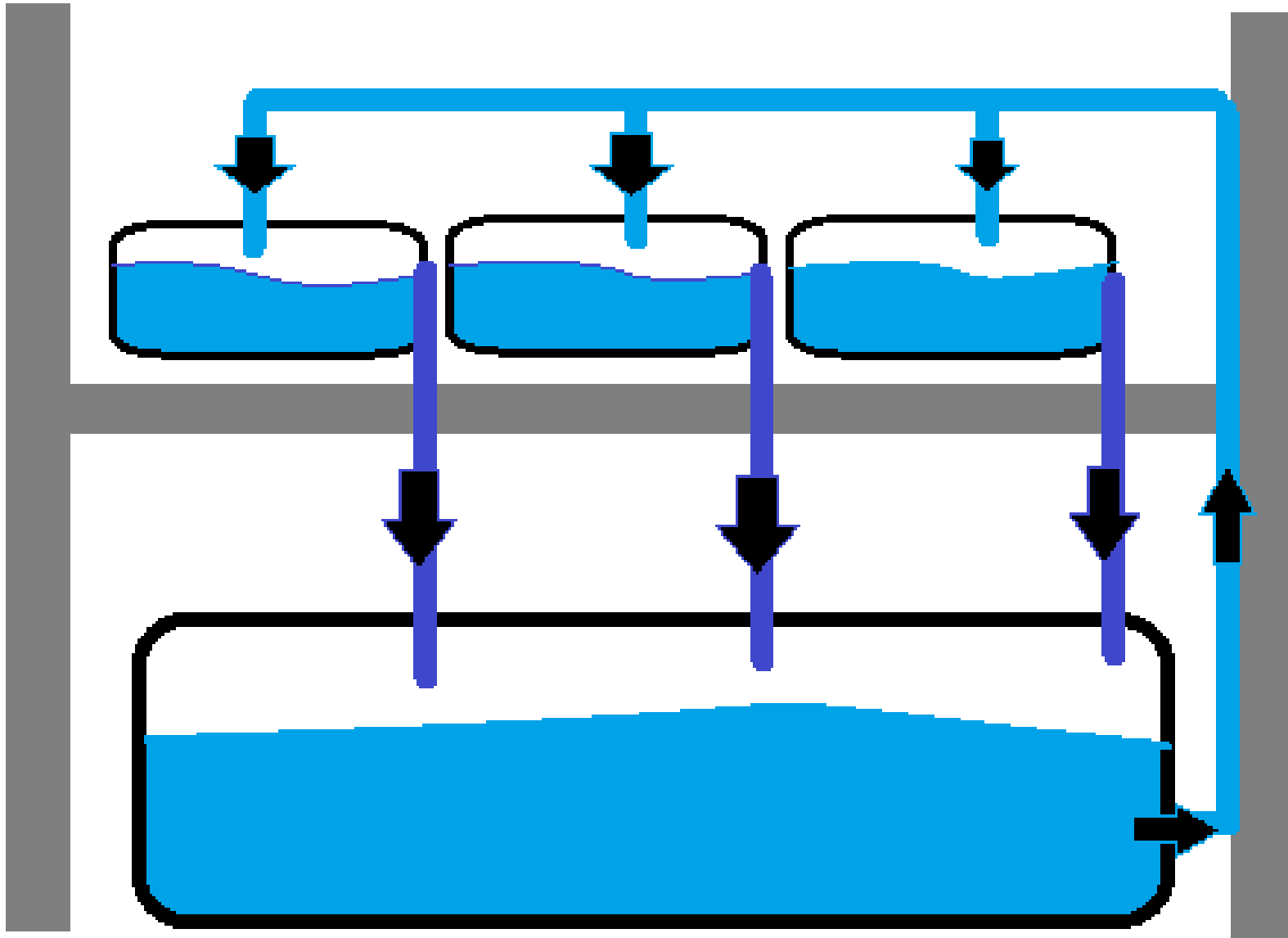


Figure 2. Study area around Robinson, Illinois.



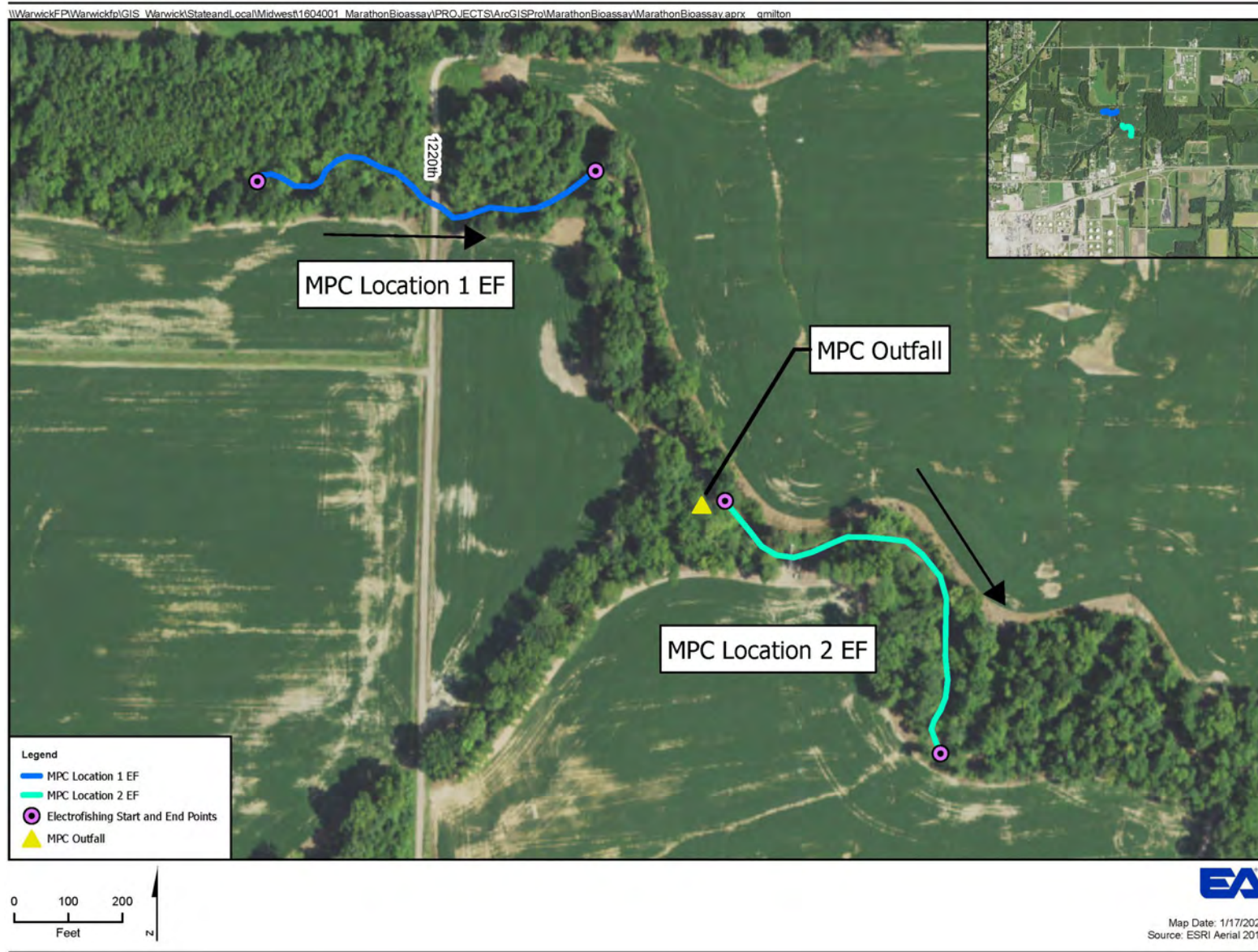
**Figure 3.** Modified flow through system test design for the onsite thermal bioassay.



**Figure 4.** Multiple views of the test setup for the onsite thermal bioassay.

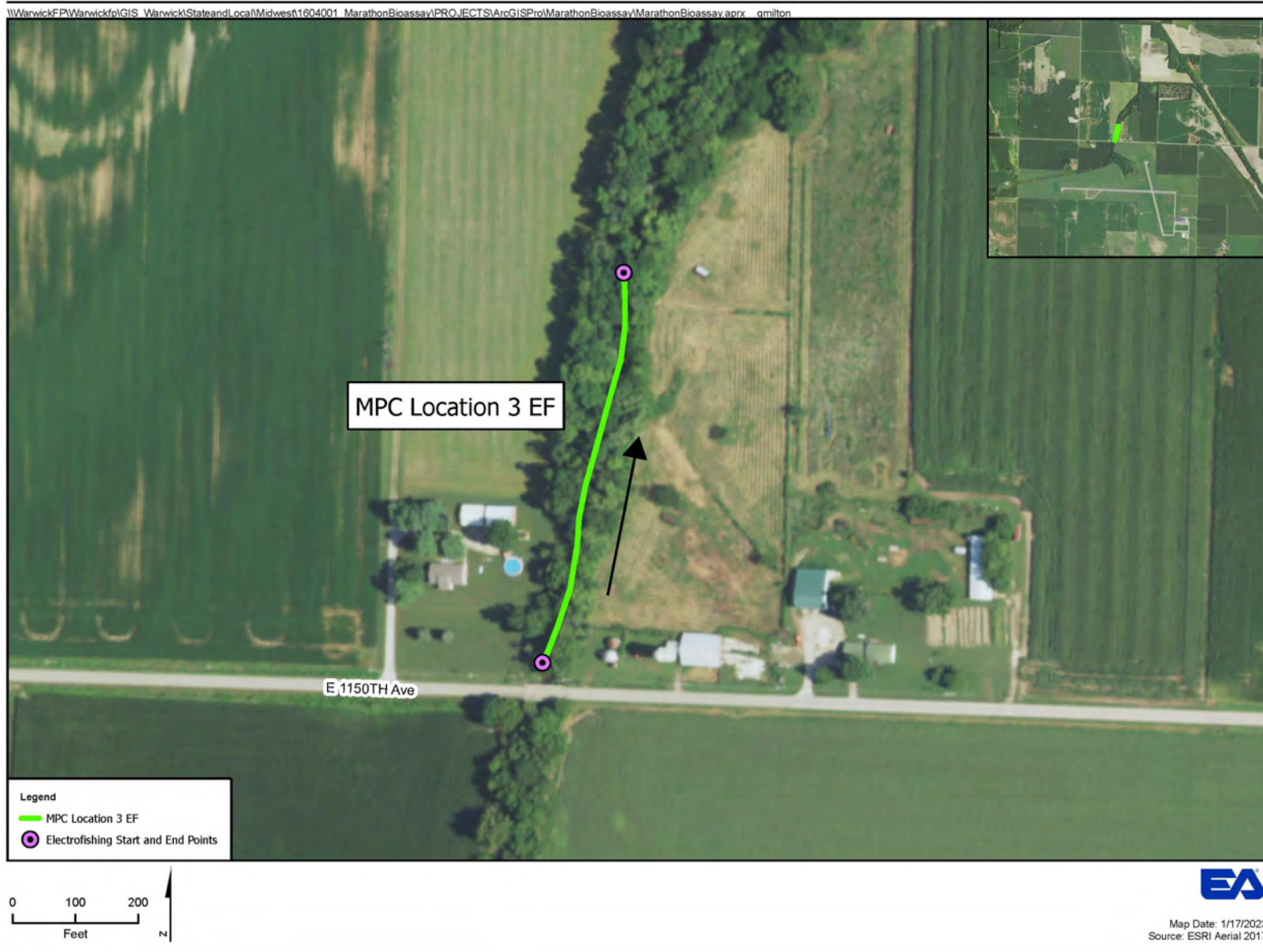


**Figure 5.** Robinson Creek sampling Locations 1 and 2 in reference to the MPC 001 outfall.





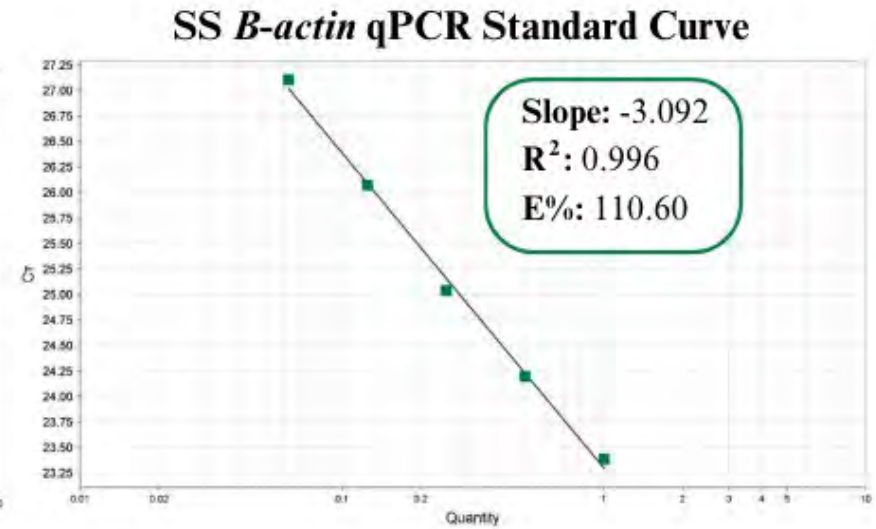
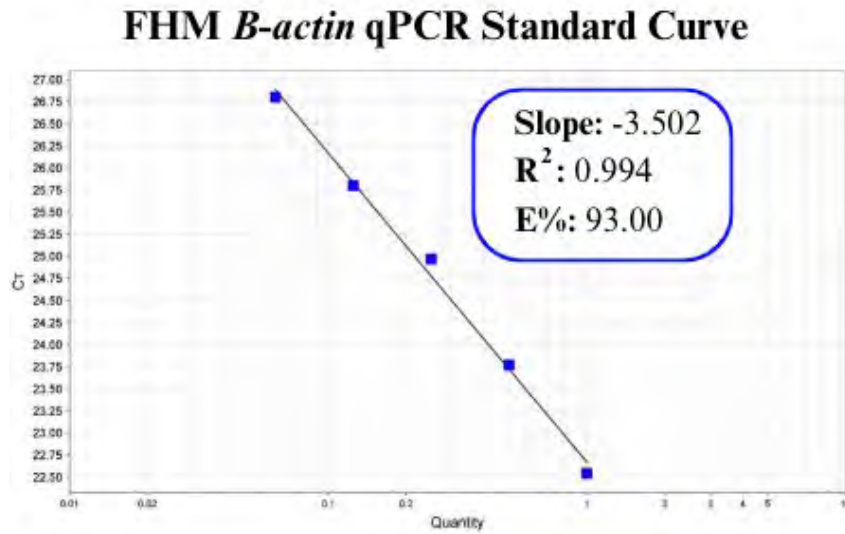
**Figure 6.** Robinson Creek downstream sampling Location 3 approximately 1.0 RM upstream of the confluence with Sugar Creek.



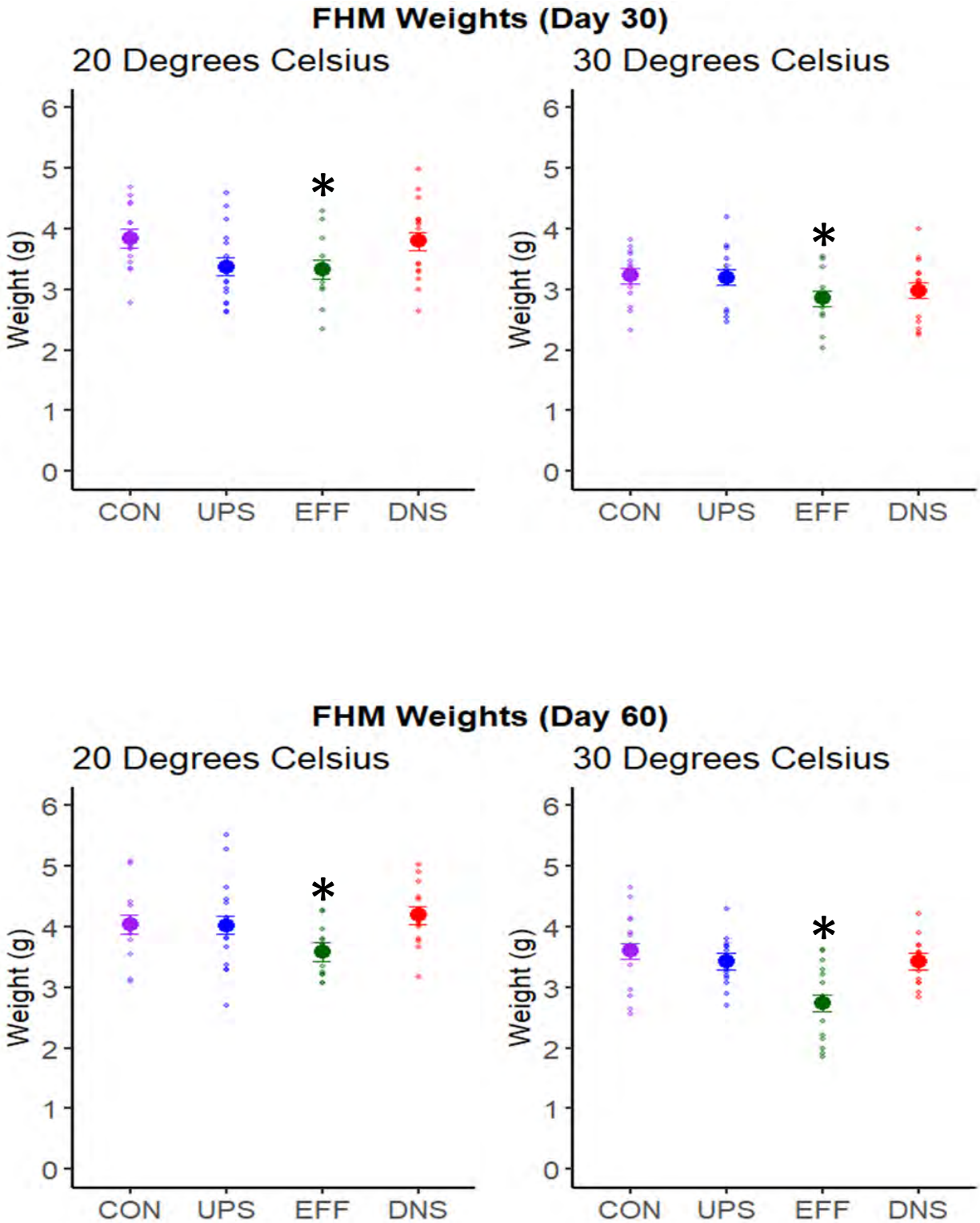
**Figure 7.** Thermal gradient for verification of fathead minnow  $\beta$ -actin primer. Tested temperatures are, from left to right, 60.0°C, 59.2°C, 58.0°C, 56.1°C, 53.8°C, 51.9°C, 50.7°C, and 50.0°C which correspond to lanes 1 through 8, respectively.



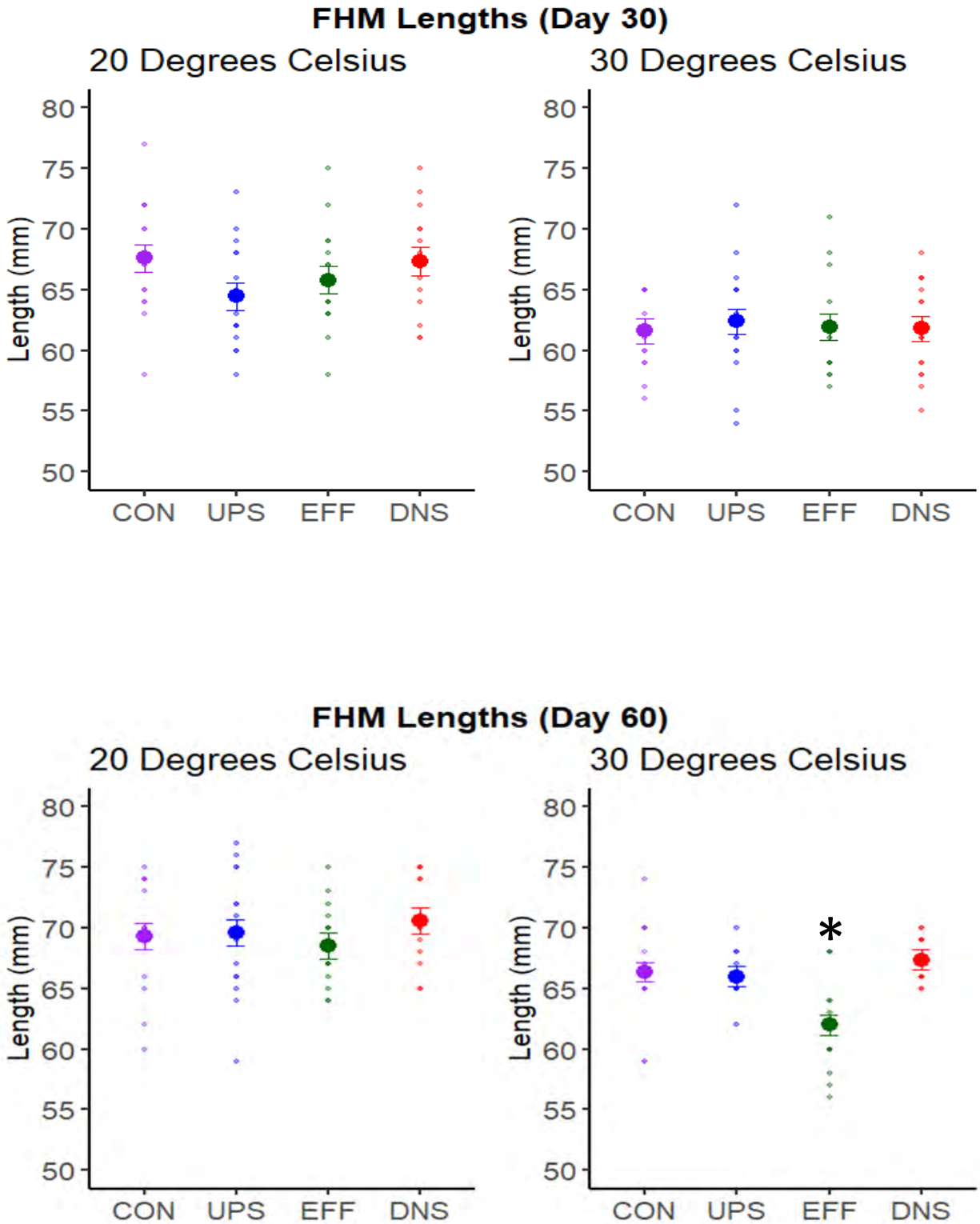
**Figure 8.** Standard curve for validation of Fathead Minnow and Spottfin Shiner *β-actin* primer. E% represents the primer efficiency.



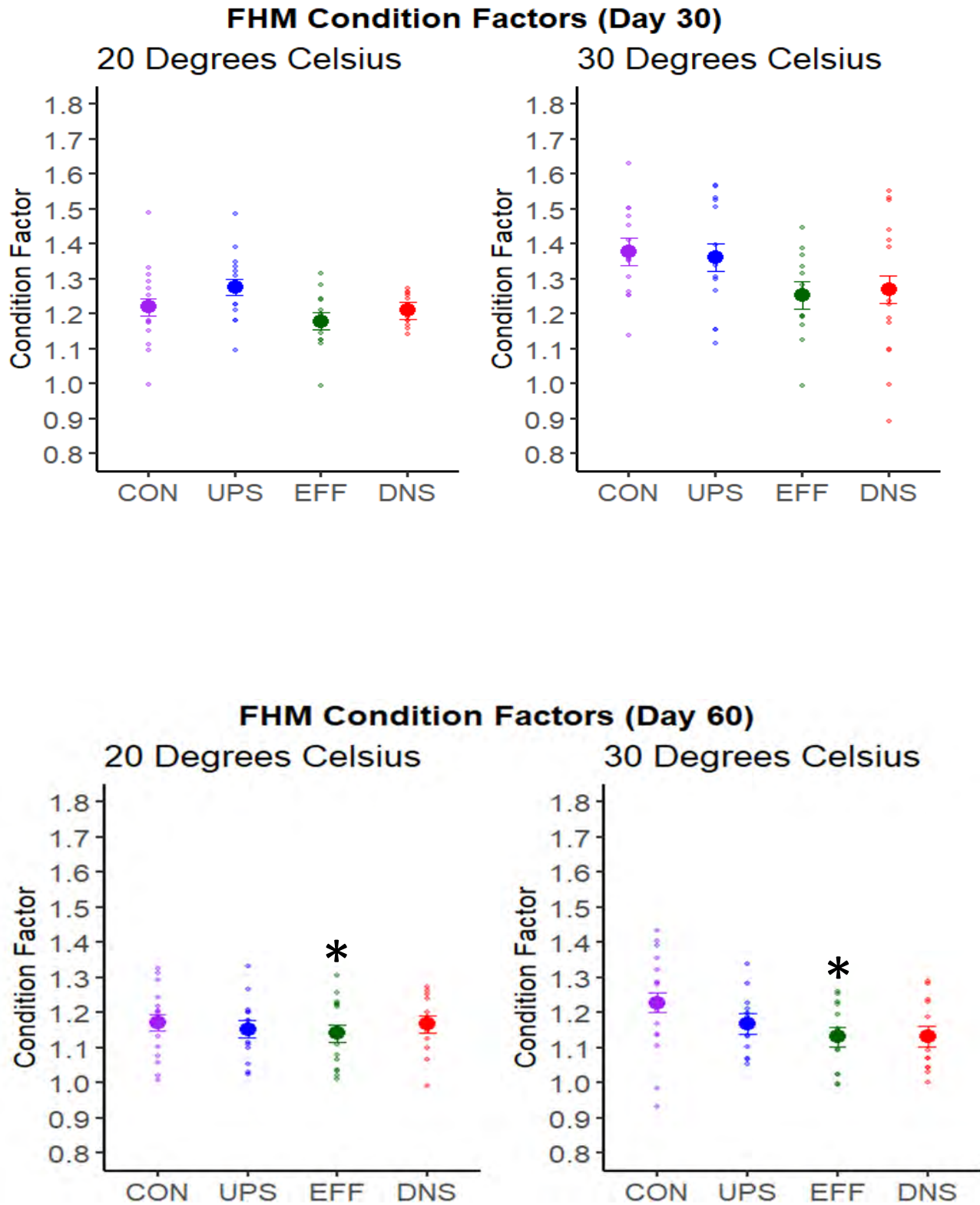
**Figure 9.** Body weight in Fathead Minnow males exposed to different water treatments (CON, UPS, EFF; and DNS) for Day 30 (top) and Day 60 (bottom). Raw data displayed in the background. Dots represent treatment mean  $\pm$  SE. \* = statistical significance ( $p \leq 0.05$ ) based on post-hoc Dunnett test on water treatment.  $n = 13-15$  per water treatment.



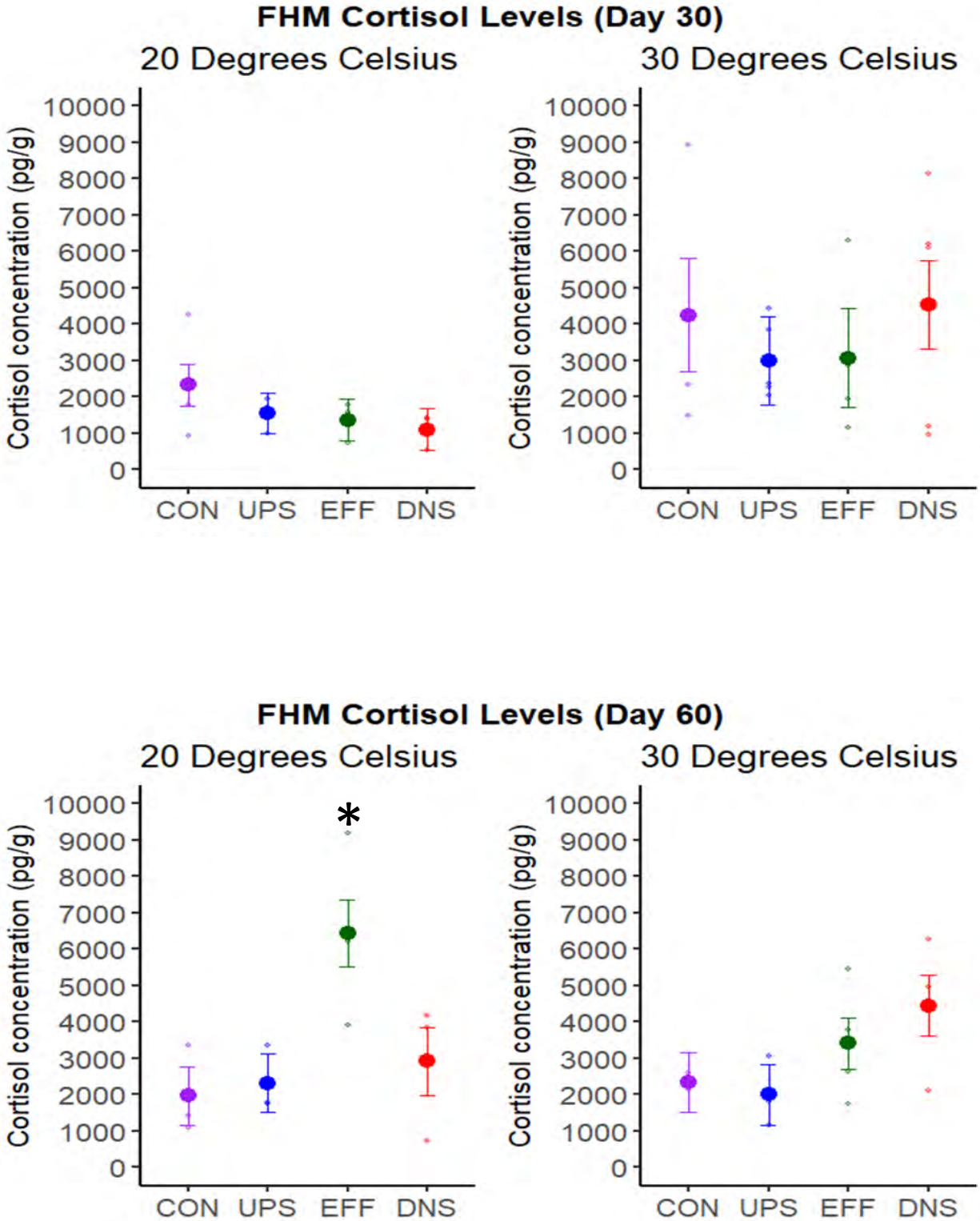
**Figure 10.** Total length in Fathead Minnow males exposed to different water treatments (CON, UPS, EFF; and DNS) for Day 30 (top) and Day 60 (bottom). Raw data displayed in the background. Dots represent treatment mean  $\pm$  SE. \* = marginal statistical significance ( $p < 0.06$ ) based on post-hoc Dunnett test on water treatment.  $n = 13-15$  per water treatment.



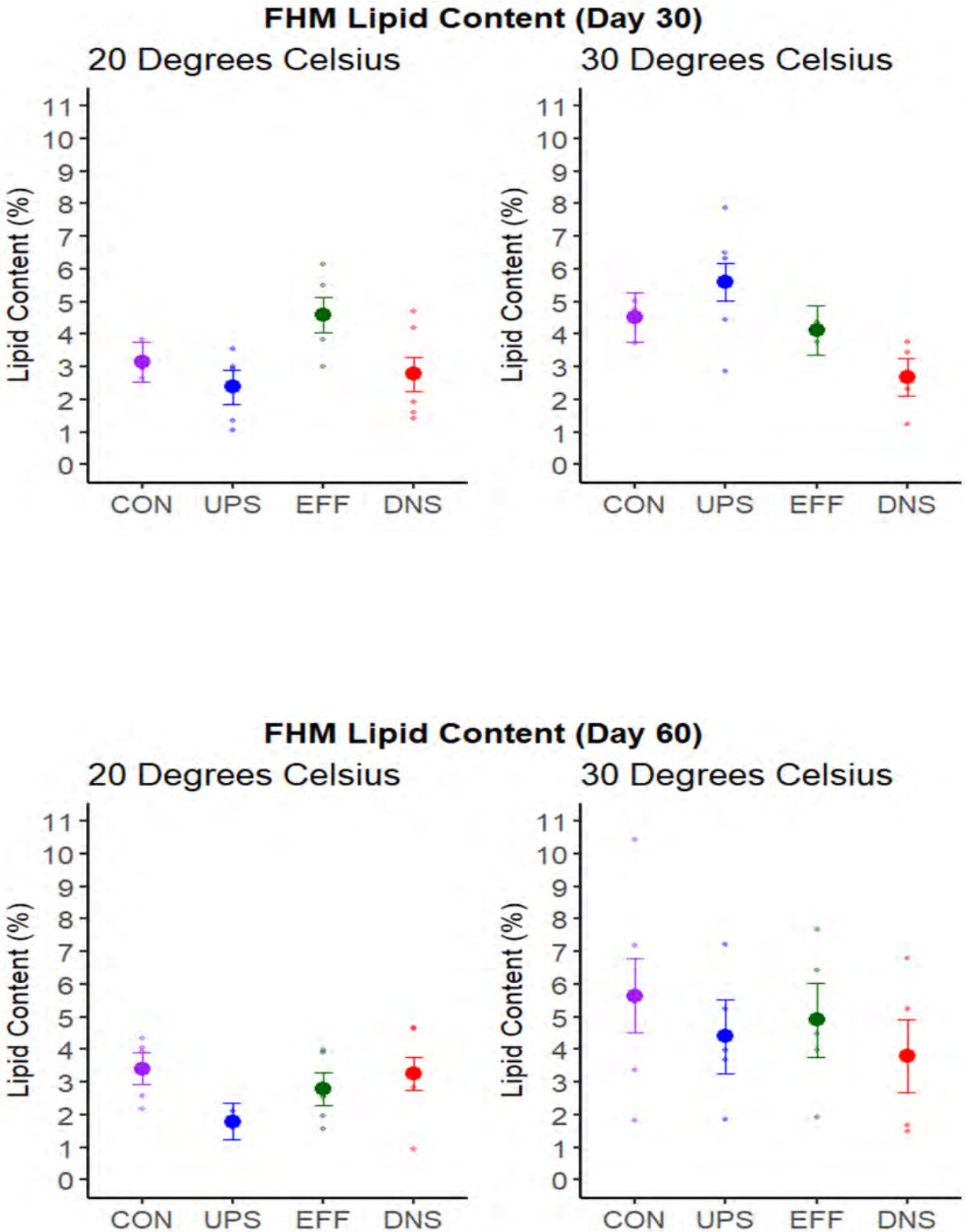
**Figure 11.** Condition Factor in Fathead Minnow males exposed to different water treatments (CON, UPS, EFF; and DNS) for Day 30 (top) and Day 60 (bottom). Raw data displayed in the background. Dots represent treatment mean  $\pm$  SE. n= 13-15 per water treatment.



**Figure 12.** Fin cortisol concentrations in Fathead Minnow males exposed to different water treatments (CON, UPS, EFF; and DNS) for Day 30 (top) and Day 60 (bottom). Raw data displayed in the background. Dots represent treatment mean  $\pm$  SE. \* = statistical significance ( $p \leq 0.05$ ) based on post-hoc Dunnett test on water treatment.  $n= 3-5$  per water treatment.

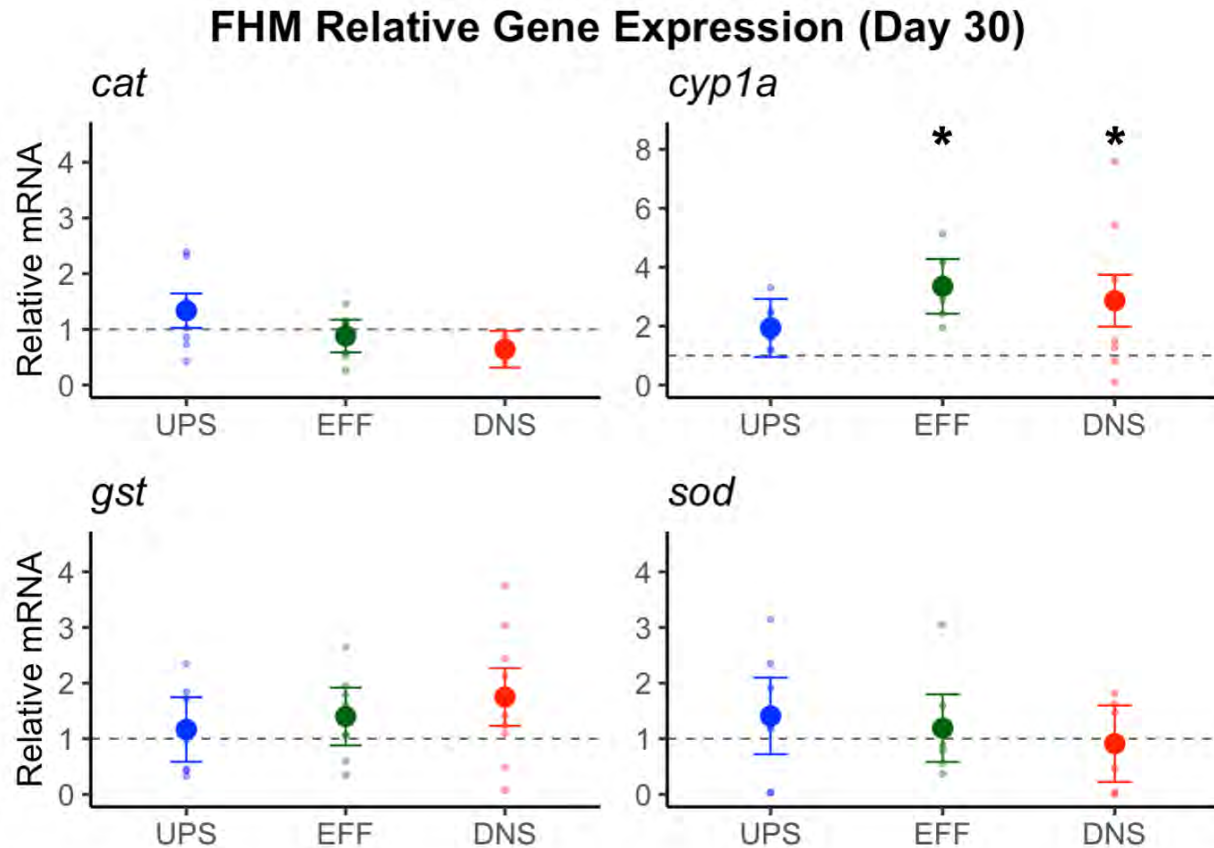


**Figure 13.** Percent lipid content in livers from Fathead Minnow males exposed to different water treatments (CON, UPS, EFF; and DNS) for Day 30 (top) and Day 60 (bottom). Raw data displayed in the background. Dots represent treatment mean  $\pm$  SE. n= 3-5 per water treatment.

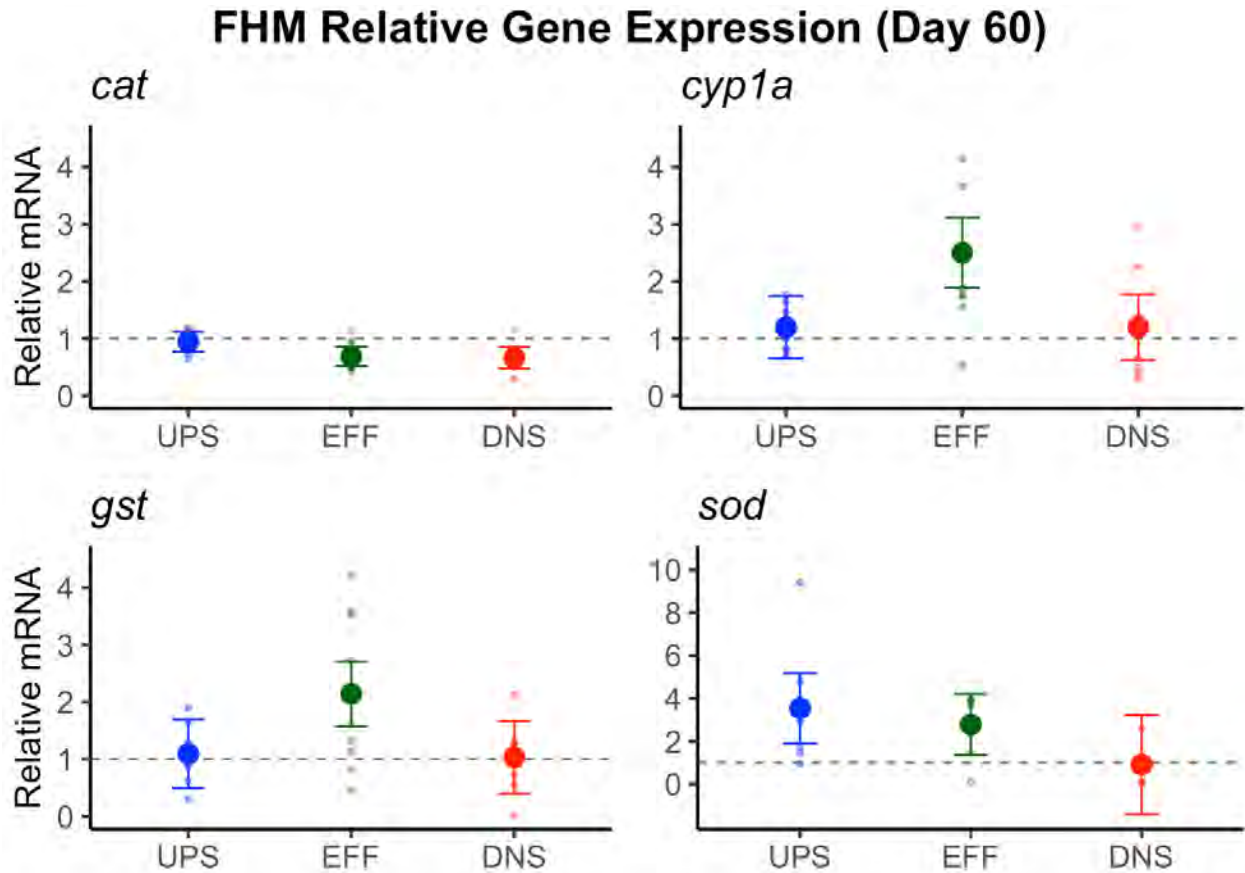




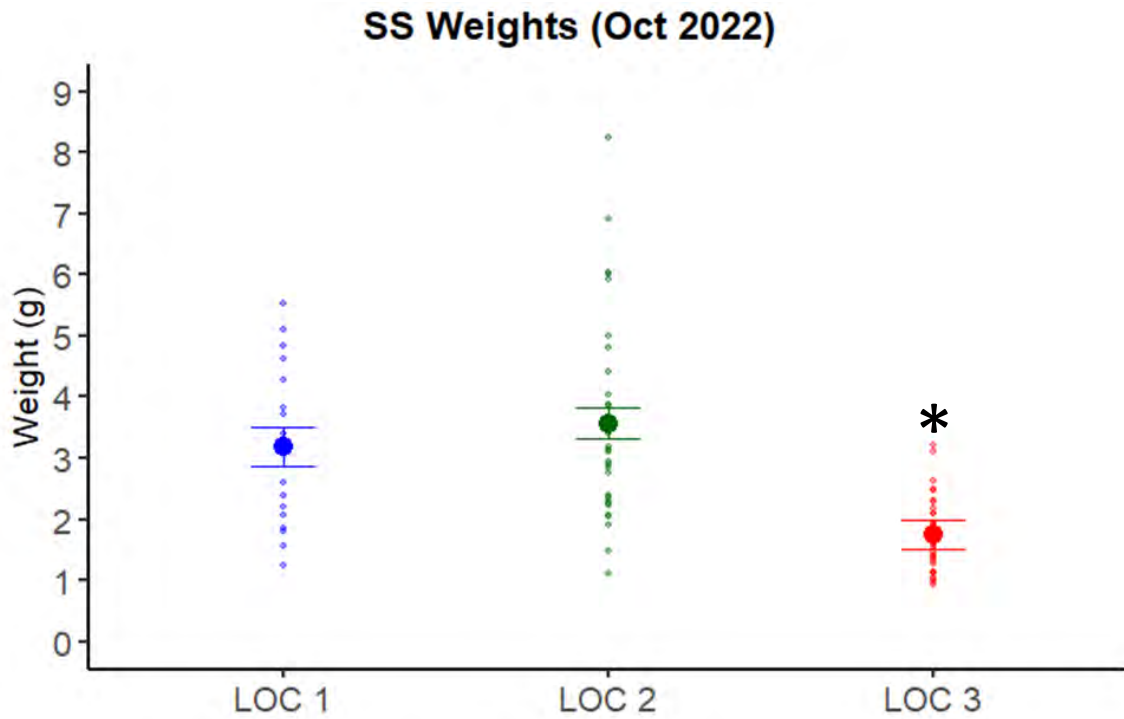
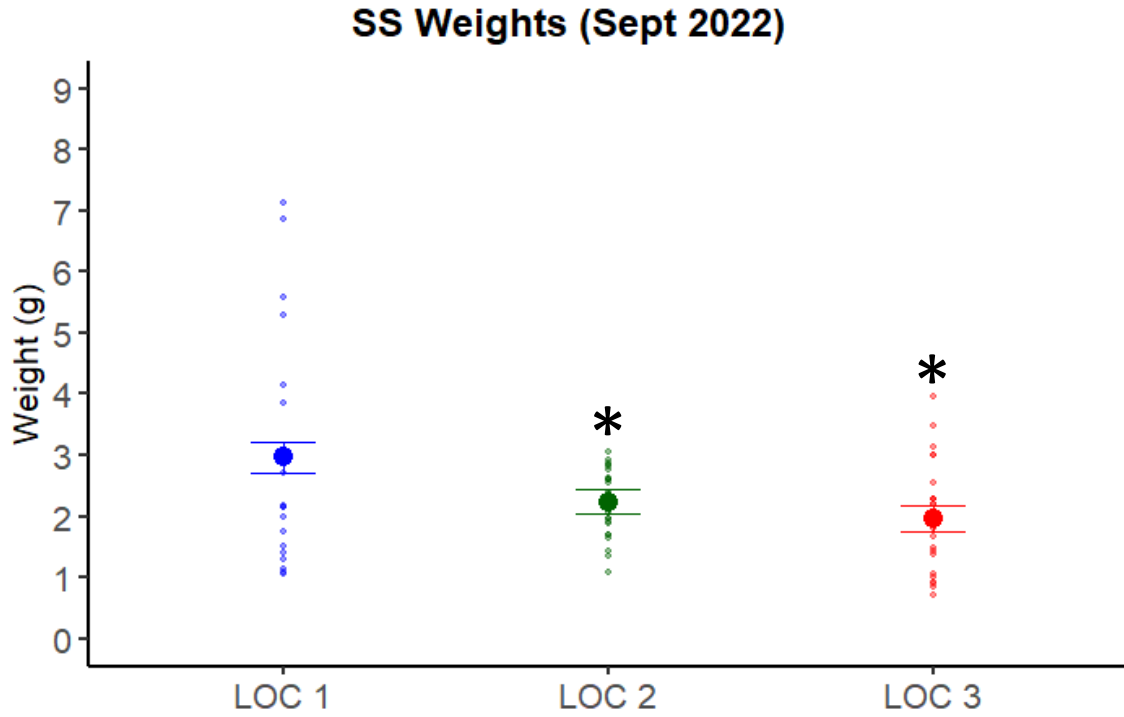
**Figure 14.** Oxidative stress target genes (*cat*, *cyp1a*, *gst*, *sod*) expression in livers of Fathead Minnow males exposed to different water treatments (UPS, EFF; and DNS) for Day 30. Values relative to controls represented by the dashed line at 1.0. Raw data displayed in the background. Dots represent treatment mean  $\pm$  95% CI. \* = statistical significance ( $p \leq 0.05$ ) based on post-hoc Dunnett test on water treatment.  $n = 7-10$  per water treatment.



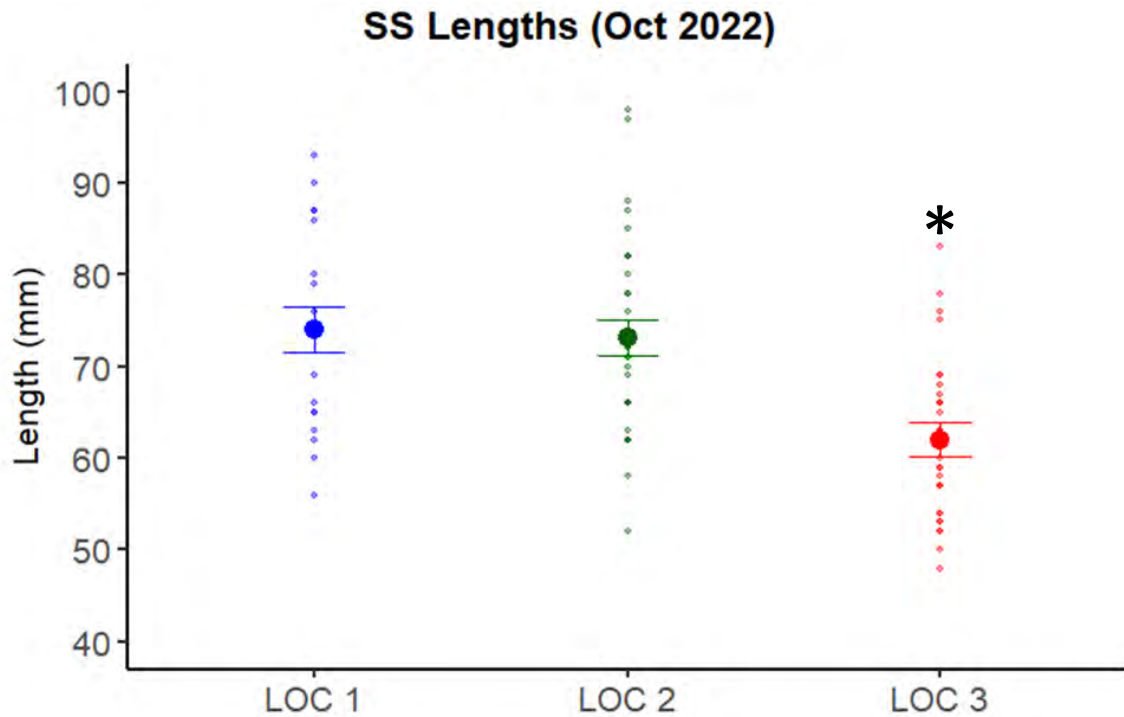
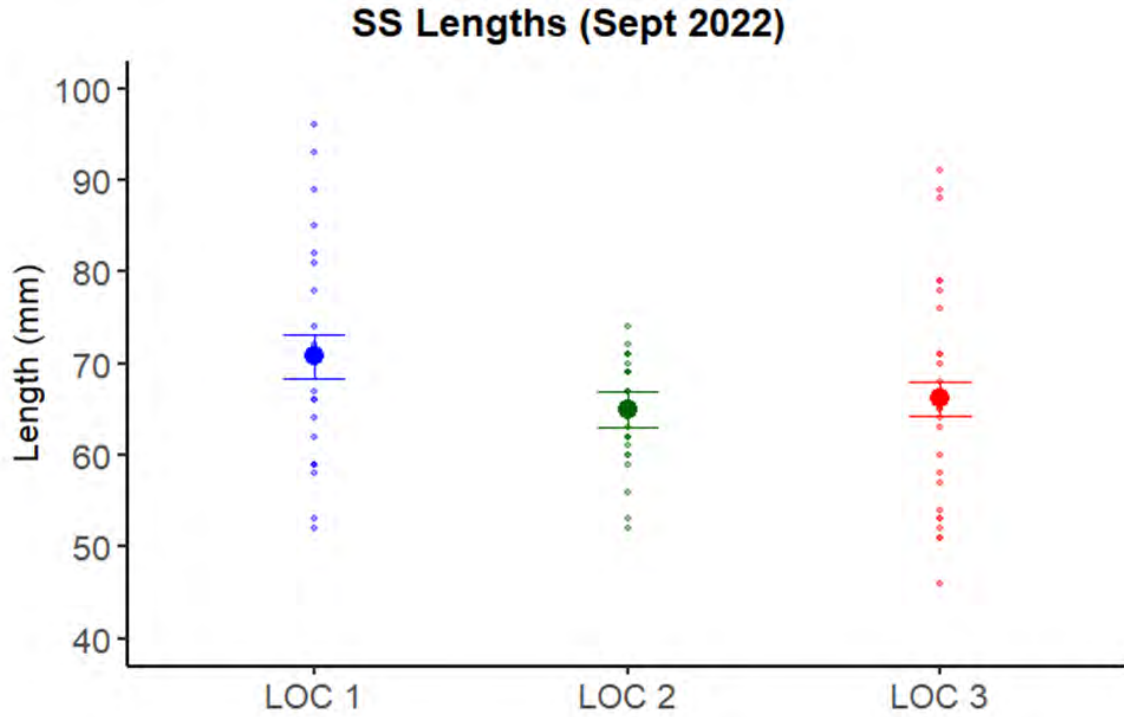
**Figure 15.** Oxidative stress target genes (*cat*, *cyp1a*, *gst*, *sod*) expression in livers of Fathead Minnow males exposed to different water treatments (UPS, EFF; and DNS) for Day 60. Values relative to controls represented by the dashed line at 1.0. Raw data displayed in the background. Dots represent treatment mean  $\pm$  95% CI. \* = statistical significance ( $p \leq 0.05$ ) based on post-hoc Dunnett test on water treatment. n= 7-10 per water treatment.



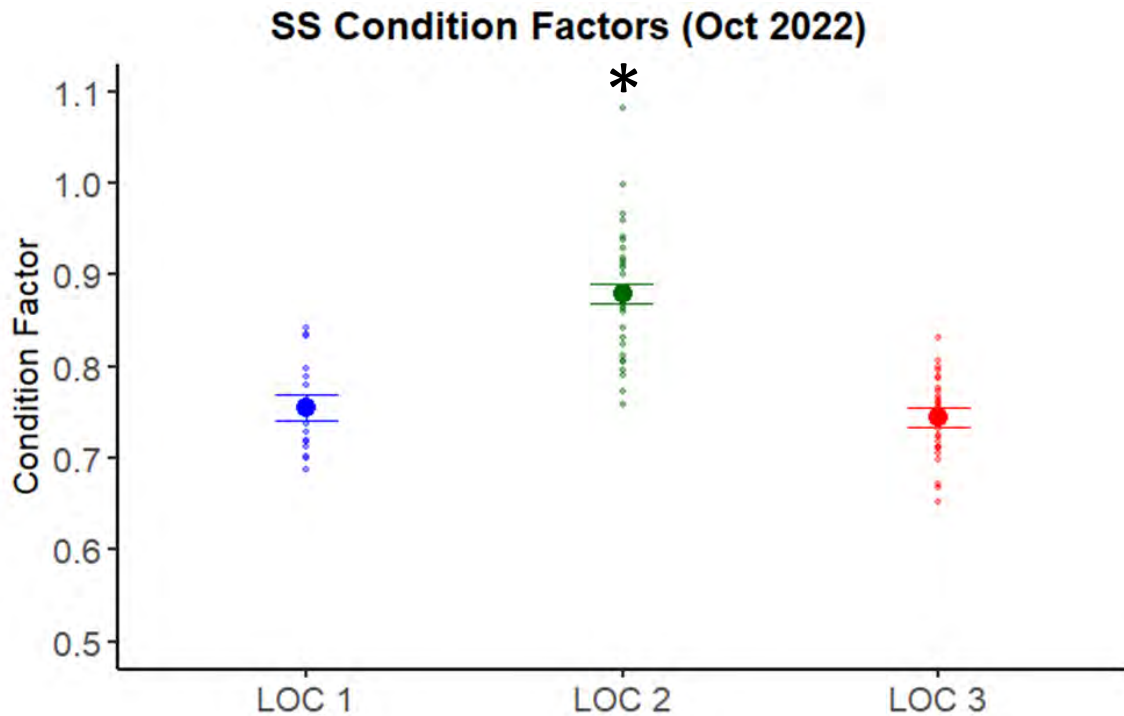
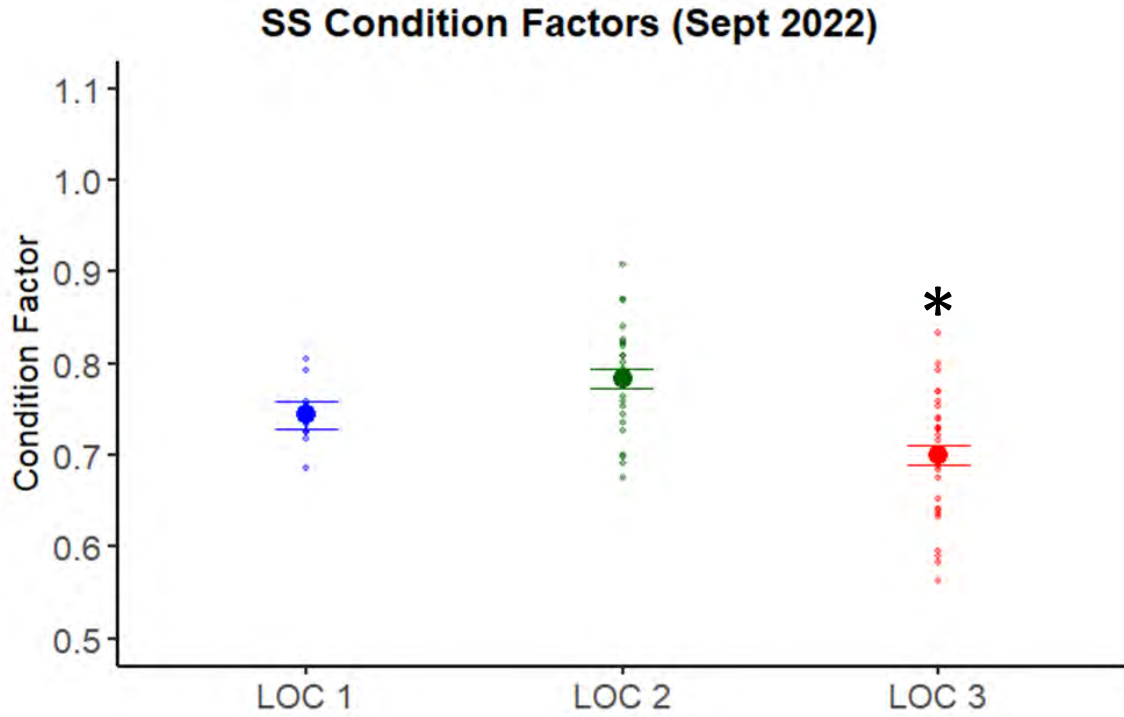
**Figure 16.** Body weight in Spotfin Shiner sampled at different field sites along Robinson Creek in September (Day 30) and October (Day 60) at Location 1, 2, and 3. Raw data displayed in the background. Dots represent treatment mean  $\pm$  SE. \* = statistical significance ( $p \leq 0.05$ ) based on post-hoc Dunnett test on water treatment.  $n = 17-30$  per water treatment.



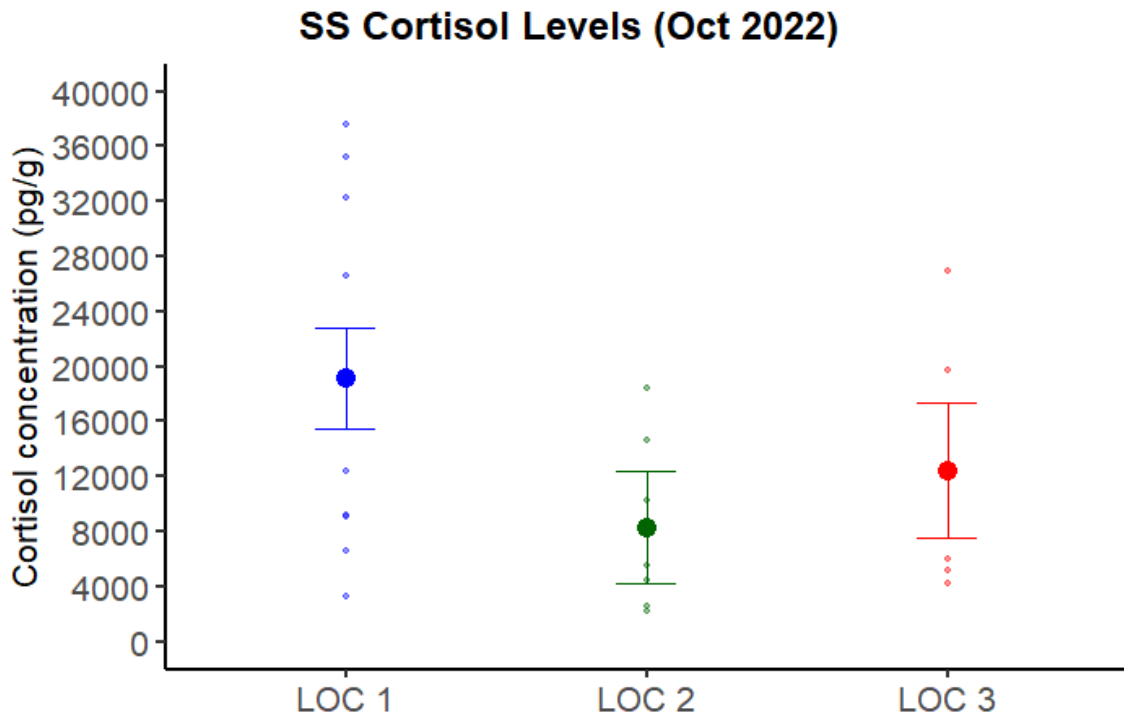
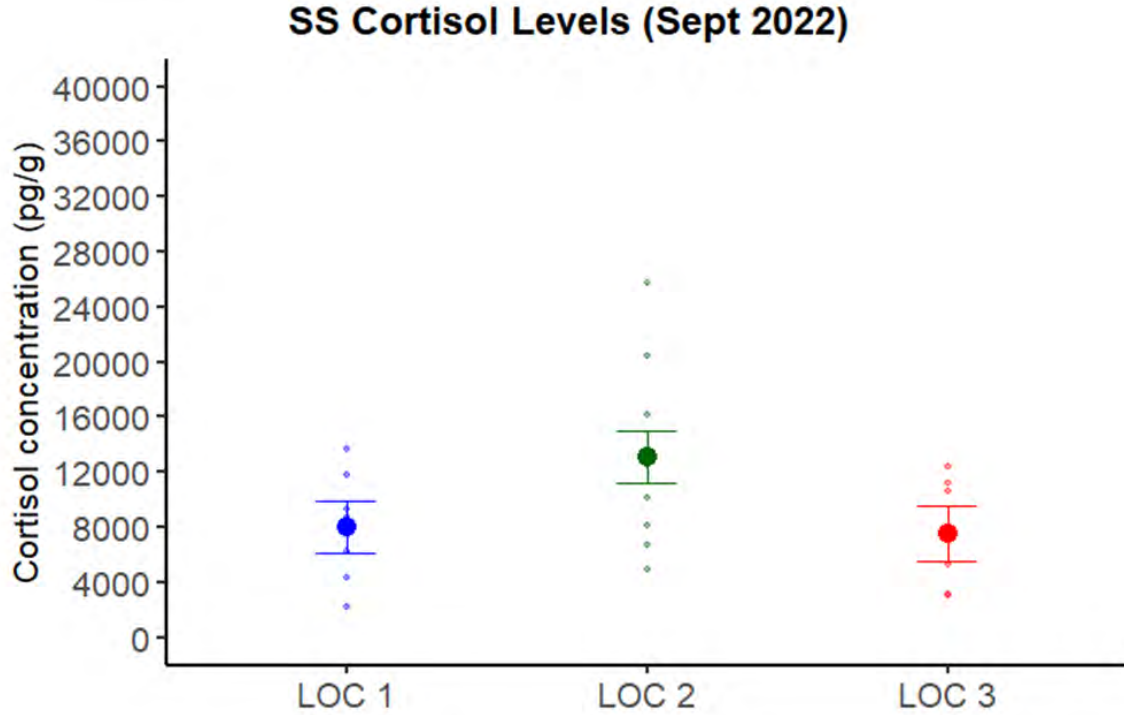
**Figure 17.** Total length in Spotfin Shiner sampled at different field sites along Robinson Creek in September (Day 30) and October (Day 60) at Location 1, 2, and 3. Raw data displayed in the background. Dots represent treatment mean  $\pm$  SE. \* = statistical significance ( $p \leq 0.05$ ) based on post-hoc Dunnett test on water treatment.  $n = 17-30$  per water treatment.



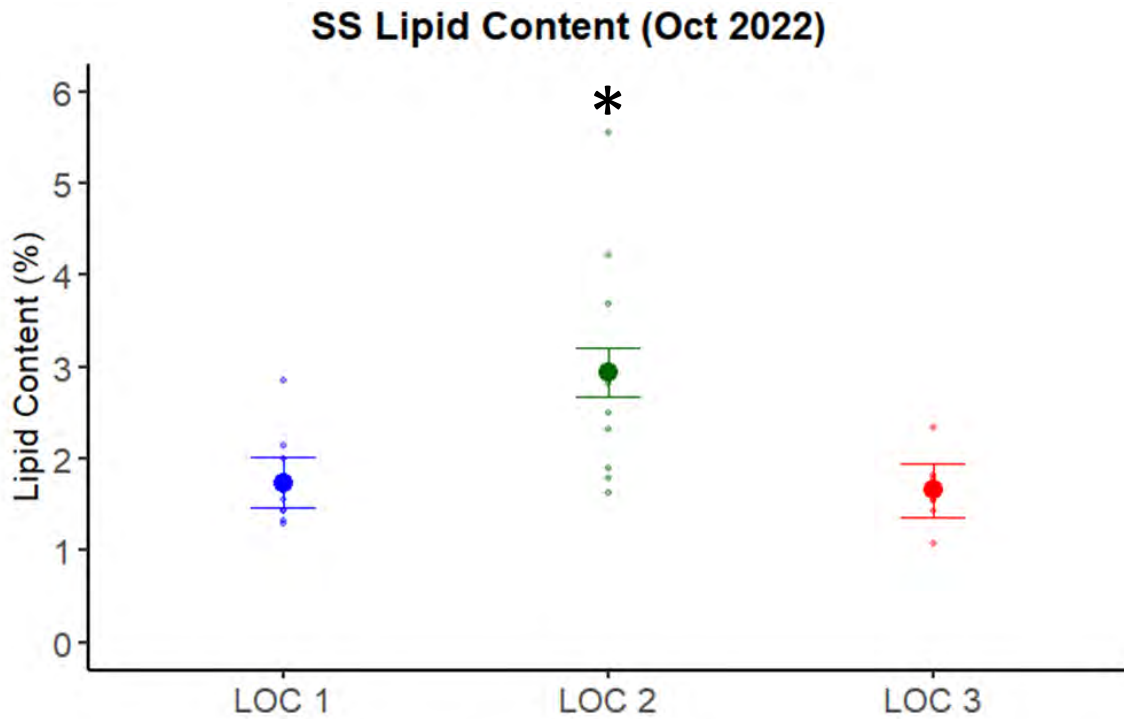
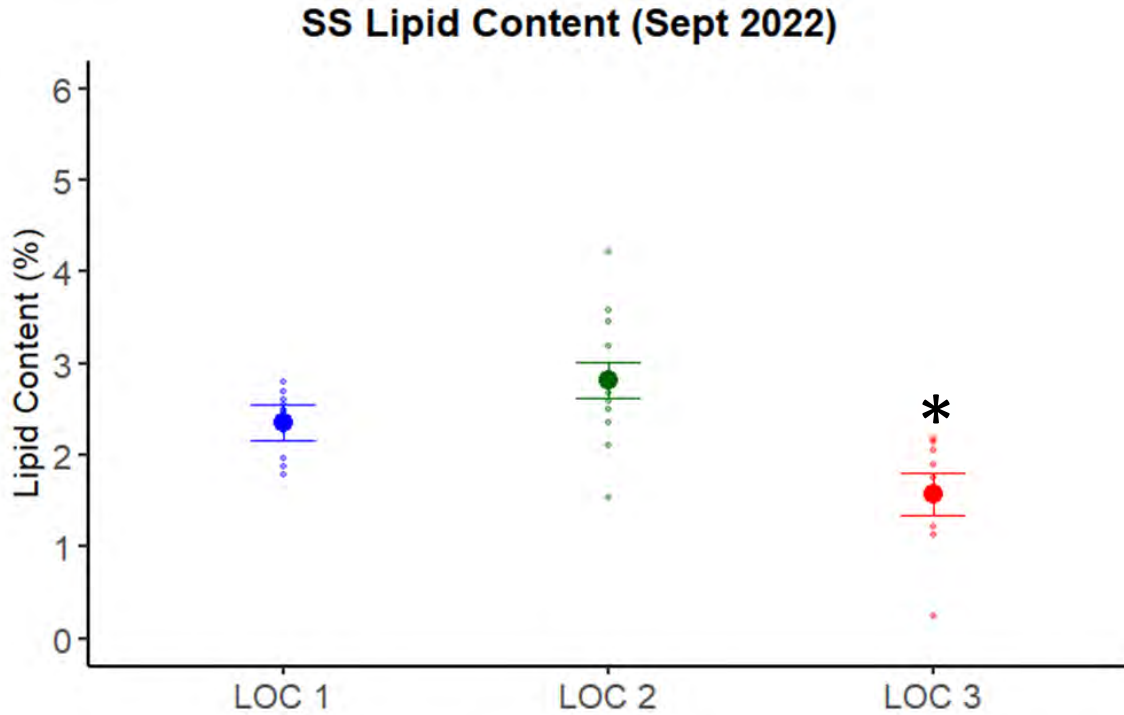
**Figure 18.** Condition Factor in Spotfin Shiner sampled at different field sites along Robinson Creek in September (Day 30) and October (Day 60) at Location 1, 2, and 3. Raw data displayed in the background. Dots represent treatment mean  $\pm$  SE. \* = statistical significance ( $p \leq 0.05$ ) based on post-hoc Dunnett test on water treatment.  $n = 17-30$  per water treatment.



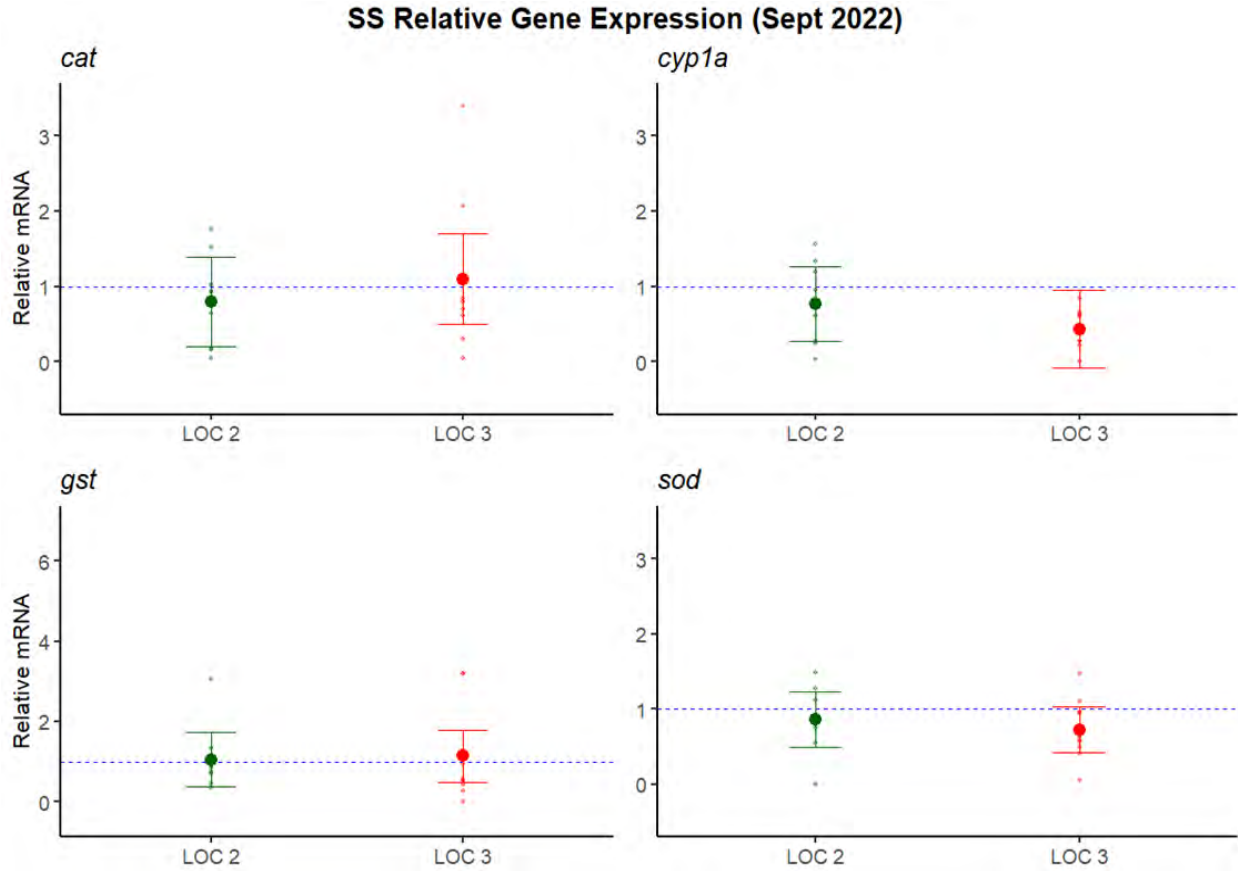
**Figure 19.** Fin cortisol concentrations in Spotfin Shiner sampled at different field sites along Robinson Creek in September (Day 30) and October (Day 60) at Location 1, 2, and 3. Raw data displayed in the background. \* = statistical significance ( $p \leq 0.05$ ) based on post-hoc Dunnett test on water treatment.  $n= 5-9$  per water treatment.



**Figure 20.** Percent (%) lipids in livers from SS sampled at different field sites along Robinson Creek in September (left panel) or October (right panel). (Upstream, UPS; Effluent, EFF; Downstream, DPS). Raw data displayed in the background. Dots represent treatment mean  $\pm$  SE. \* = statistical significance ( $p \leq 0.05$ ) based on post-hoc Dunnett test on water treatment.  $n=8-10$  per water treatment.

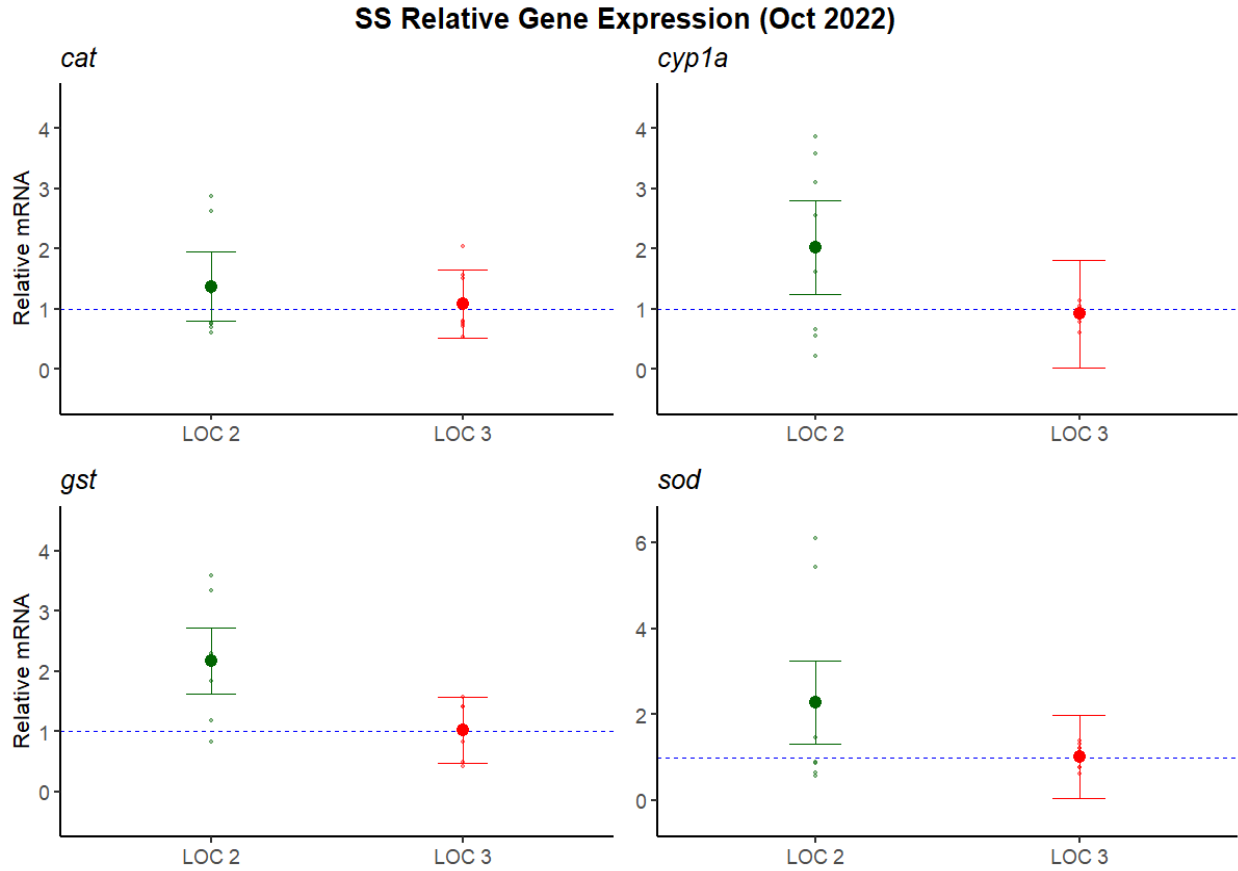


**Figure 21.** Oxidative stress target genes (*cat*, *sod*, *cyp1a*, *gst*) expression in livers of SS sampled at different field sites along Robinson Creek in September 2022. Values relative to Location 1 represented by the dashed line at 1.0. Raw data displayed in the background. Dots represent treatment mean  $\pm$  95% CI. n= 7-10 per treatment.





**Figure 22.** Oxidative stress target genes (*cat*, *sod*, *cyp1a*, *gst*) expression in livers of SS sampled at different field sites along Robinson Creek in October 2022. Values relative to Location 1 represented by the dashed line at 1.0. Raw data displayed in the background. Dots represent treatment mean  $\pm$  95% CI. n= 7-10 per treatment.



## **Tables**

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**Table 1.** Water chemistry analytes measured on Day 0, 30, and 60 of the study.

1. Acenaphthene	44. Methylene chloride	87. Dieldrin
2. Acrolein	45. Bromoform	88. Chlordane
3. Acrylonitrile	46. Bromomethane	89. 4,4-DDT
4. Benzene	47. Chloromethane	90. 4,4-DDE
5. Benzidine	48. Dichlorobromomethane	91. 4,4-DDD
6. Carbon tetrachloride	49. Bromodichloromethane	92. Alpha-endosulfan
7. Chlorobenzene	50. Hexachloro-1,3,-butadiene	93. Beta-endosulfan
8. 1,2,4-trichlorobenzene	51. Hexachlorocyclopentadiene	94. Endosulfan sulfate
9. Hexachlorobenzene	52. Isophorone	95. Endrin
10. 1,2-dichloroethane	53. Naphthalene	96. Endrin aldehyde
11. 1,1,1-trichloroethane	54. Nitrobenzene	97. Heptachlor
12. Hexachloroethane	55. 2-nitrophenol	98. Heptachlor epoxide
13. 1,1-dichloroethane	56. 4-nitrophenol	99. Alpha-BHC
14. 1,1,2-trichloroethane	57. 2,4-dinitrophenol	100. Beta-BHC
15. 1,1,2,2-tetrachloroethane	58. N-nitrosodimethylamine	101. Gamma-BHC
16. Chloroethane	59. N-nitrosodiphenylamine	102. Delta-BHC
17. Bis(2-chloroethyl) ether	60. N-nitrosodi-n-propylamine	103. PCB-1242 (Arochlor 1242)
18. 2-chloroethyl vinyl ethers	61. Pentachlorophenol	104. PCB-1254 (Arochlor 1254)
19. 2-chloronaphthalene	62. Phenol	105. PCB-1221 (Arochlor 1221)
20. 2,4,6-trichlorophenol	63. Bis(2-ethylhexyl) phthalate	106. PCB-1232 (Arochlor 1232)
21. Chloroform	64. Butyl benzyl phthalate	107. PCB-1248 (Arochlor 1248)
22. 2-chlorophenol	65. Di-n-Butyl Phthalate	108. PCB-1260 (Arochlor 1260)
23. 1,2-dichlorobenzene	66. Di-n-octyl phthalate	109. PCB-1016 (Arochlor 1016)
24. 1,3-dichlorobenzene	67. Diethyl Phthalate	110. Toxaphene
25. 1,4-dichlorobenzene	68. Dimethyl phthalate	111. Antimony
26. 3,3-dichlorobenzidine	69. Benzo(a) anthracene	112. Arsenic
27. 1,1-dichloroethylene	70. Benzo(a) pyrene	113. Beryllium
28. 1,2-trans-dichloroethylene	71. Benzo(b) fluoranthene	114. Cadmium
29. 2,4-dichlorophenol	72. Benzo(k) fluoranthene	115. Chromium
30. 1,2-dichloropropane	73. Chrysene	116. Copper
31. 1,3-cis-dichloropropene	74. Acenaphthylene	117. Cyanide, Total
32. 1,3-trans-dichloropropene	75. Anthracene	118. Lead
33. 2,4-dimethylphenol	76. Benzo(ghi) perylene	119. Mercury
34. 4,6-dinitro-2-methylphenol	77. Fluorene	120. Nickel
35. 2,4-dinitrotoluene	78. Phenanthrene	121. Selenium
36. 2,6-dinitrotoluene	79. Dibenzo(a,h) anthracene	122. Silver
37. 1,2-diphenylhydrazine	80. Indeno (1,2,3-cd) pyrene	123. Thallium
38. Ethylbenzene	81. Pyrene	124. Zinc
39. Fluoranthene	82. Tetrachloroethylene	125. 2,3,7,8-TCDD
40. 4-chlorophenyl phenyl ether	83. Toluene	
41. 4-bromophenyl phenyl ether	84. Trichloroethylene	
42. Bis(2-chloroisopropyl) ether	85. Vinyl chloride	
43. Bis(2-chloroethoxy) methane	86. Aldrin	

**Table 2.** Description and river mile (RM) of Robinson Creek sampling locations.

<b>Location</b>	<b>RM</b>	<b>Description</b>	<b>Latitude / Longitude Start</b>	<b>Latitude / Longitude End</b>
1	5.2	Ambient conditions, upstream of the MPC thermal discharge (Outfall 001) and downstream of the Robinson Publicly Owned Treatment Works (POTW). This location was the same as RC04 in the 2016 316(a) Demonstration (MBI 2016).	39.0146670117974 / -87.7085305843502	39.0145956818014 / -87.7107545547187
2	5.0	Near-field location beginning immediately downstream of the MPC thermal discharge. This location was the same as MPMZ in the 2016 316(a) Demonstration (MBI 2016)	39.0117316693067 / -87.706283563748	39.0130019467324 / -87.7076980099081
3	1.0	Far-field location within the lower reaches of Robinson Creek. This location was the same as RC09 in the 2016 316(a) Demonstration (MBI 2016)	39.024241156876 / -87.6522405724972	39.0225344337522 / -87.6526650320738

**Table 3.** RT-qPCR primers for FHM and SS for target genes of interest (*cat*, *sod*, *cyp1a*, & *gst*) and reference gene (*b-actin*). ‘TA’ = Annealing Temperature; ‘E%’ = Efficiency calculated from slope of standard curve; ‘[Primer]’ = Concentration of each primer (F & R) per qPCR reaction. “\_d” = indicates a degenerate primer design.

Target Gene	F/Left Primer (5'-3')	R/Right Primer (5'-3')	T <sub>A</sub> (°C)	Species	E%	[Primer]	Amplicon	NCBI Accession	Source	DOI
<i>b-actin</i>	AAGATCTGGCATCACACCTTCT	ACCTGTGTCATCTTTCCCTGT	57.1	FHM; SS	93.0 110.6	187.5 nM 250 nM	116 bp	DQ447717.1	Self-designed	NA
<i>cat</i>	GACCGAGAGAGGATAACCAGAGA	TTGGCTTTACAATAGCGTCTGA	58.2	FHM	85.0	125 nM	101 bp	XM_039669564.1	Self-designed	NA
<i>sod</i>	CCAGACATGTCGGAGACCTT	ATGGAATGTTGCCCTGAGAG	57.0 49.7	FHM; SS	90.4 97.9	125 nM 187.5 nM	NA	NA (unpublished)	(He et al. 2012)	<a href="https://doi.org/10.1016/j.watres.2012.09.004">https://doi.org/10.1016/j.watres.2012.09.004</a>
<i>gst</i>	GGAAGTGTTTTTGACCAAGAGG	AGGTTGTATTTCCAGCGATGT	56.0	FHM	95.3	250 nM	150 bp	EF628373.1	Self-designed	NA
<i>cyp1a</i>	GCAGGGAGAAGTGGAGAGAGAAG	GACGTACAGTGAGGAATGGTGA	57.1	FHM	92.4	125 nM	144 bp	XM_039680280.1	Self-designed	NA
<i>cat_d</i>	GACCGAGAGMGGATAACCAGAGA	TTGGCTTTRSARTAGCGYSTGA	53.7	SS	89.4	250 nM	101 bp	XM_039669564.1	Self-designed	NA
<i>gstt_d</i>	ATCTCTGGCTGATCTTGTRGCC	GACTTCCACRCCRATCTCCTTC	58.1	SS	107.6	187.5 nM	130 bp	XM_039680288.1	Self-designed	NA
<i>Cyp1a_d</i>	GCAGRGAGARCTGARWGARAAG	GACGTACAGTGAGGAATGGTGA	58.0	SS	98.8	187.5 nM	144 bp	XM_039680280.1	Self-designed	NA

**Table 4.** Results of Onsite Thermal Bioassay testing with *Pimephales promelas*.

Sample Identification	20 Degree Test Temperature		30 Degree Test Temperature	
	30-Day Survival (percent)	60-Day Survival (percent)	30-Day Survival (percent)	60-Day Survival (percent)
CONTROL	99	97	99	99
UPSTREAM	100	100	81 <sup>(a)</sup>	77 <sup>(a)</sup>
EFFLUENT	100	100	92	89
DOWNSTREAM	100	99	100	87

<sup>(a)</sup> Significantly different ( $p < 0.05$ ) from laboratory control.

**Table 5.** Water Quality Parameters Measured During Onsite Thermal Bioassay Testing.

<b>Sample Identification</b>	<b>Mean Temperature (°C)</b>	<b>Mean pH (su)</b>	<b>Mean Dissolved Oxygen (mg/L)</b>	<b>Mean Conductivity (µs/cm)</b>
CONTROL	20.6	7.9	8.6	525
UPSTREAM	19.6	8.2	8.9	1,049
EFFLUENT	20.2	8.1	8.9	1,886
DOWNSTREAM	19.9	8.2	8.9	1,302

<b>Sample Identification</b>	<b>Mean Temperature (°C)</b>	<b>Mean pH (su)</b>	<b>Mean Dissolved Oxygen (mg/L)</b>	<b>Mean Conductivity (µs/cm)</b>
CONTROL	29.1	8.0	7.1	509
UPSTREAM	28.3	8.2	7.4	1,003
EFFLUENT	28.7	8.2	7.3	1,908
DOWNSTREAM	28.5	8.2	7.4	1,326



**Table 6.** List of Common and Scientific Names for Fish Species Collected from Robinson Creek, September and October 2022.

Common Family Name <sup>(a)</sup>	Common Name	Scientific Name
SUCKERS	RIVER CARPSUCKER	<i>Carpionodes carpio</i>
	WHITE SUCKER	<i>Catostomus commersonii</i>
	SPOTTED SUCKER	<i>Minytrema melanops</i>
	SHORthead REDHORSE	<i>Moxostoma macrolepidotum</i>
MINNOWS	CENTRAL STONEROLLER	<i>Campostoma anomalum</i>
	SPOTFIN SHINER	<i>Cyprinella spiloptera</i>
	BIGEYE CHUB	<i>Hybopsis amblops</i>
	STRIPED SHINER	<i>Luxilus chrysocephalus</i>
	REDFIN SHINER	<i>Lythrurus umbratilis</i>
	EMERALD SHINER	<i>Notropis atherinoides</i>
	SILVERJAW MINNOW	<i>Notropis buccatus</i>
	SAND SHINER	<i>Notropis stramineus</i>
	CHANNEL SHINER	<i>Notropis wickliffi</i>
	SUCKERMOUTH MINNOW	<i>Phenacobius mirabilis</i>
	BLUNTNOSE MINNOW	<i>Pimephales notatus</i>
	CREEK CHUB	<i>Semotilus atromaculatus</i>
NORTH AMERICAN CATFISHES	YELLOW BULLHEAD	<i>Ameiurus natalis</i>
PIRATE PERCHES	PIRATE PERCH	<i>Aphredoderus sayanus</i>
NEW WORLD SILVERSIDES	BROOK SILVERSIDE	<i>Labidesthes sicculus</i>
TOPMINNOWS	BLACKSTRIPE	<i>Fundulus notatus</i>
	TOPMINNOW	
LIVEBEARERS	WESTERN MOSQUITOFISH	<i>Gambusia affinis</i>
DARTERS AND PERCHES	SLOUGH DARTER	<i>Etheostoma gracile</i>
	JOHNNY DARTER	<i>Etheostoma nigrum</i>
	LOGPERCH	<i>Percina caprodes</i>
SUNFISHES	<i>Lepomis</i> HYBRID	<i>Lepomis</i> HYBRID
	GREEN SUNFISH	<i>Lepomis cyanellus</i>
	BLUEGILL	<i>Lepomis macrochirus</i>
	LONGEAR SUNFISH	<i>Lepomis megalotis</i>
	REDEAR SUNFISH	<i>Lepomis microlophus</i>
	SPOTTED BASS	<i>Micropterus punctulatus</i>
	LARGEMOUTH BASS	<i>Micropterus salmoides</i>

(a) Family arrangement follows “Eschmeyer’s Catalog of Fishes” (Van der Laan et al. 2022). Common names of families follow Page et al. (2013), except for White Basses (Moronidae) and the newly elevated cypriniform family, Minnows (Leuciscidae), which follows Metzke et al. (2022).

**Table 7.** Total Catch and Relative Abundance of Fish Collected by Longline and Pram Electrofishing by Location, September and October 2022.

Survey Period:	21-22 September							
Location:	1		2		3		Combined	
Gear:	EFLONG		EFPRAM		EFPRAM			
Species	No.	%	No.	%	No.	%	No.	%
CENTRAL STONEROLLER	68	22.8	69	11.8	--	--	137	13.3
SILVERJAW MINNOW	17	5.7	312	53.5	27	17.9	356	34.5
BIGEYE CHUB	--	--	--	--	--	--	--	--
EMERALD SHINER	--	--	--	--	8	5.3	8	0.8
STRIPED SHINER	--	--	--	--	--	--	--	--
SPOTFIN SHINER	1	0.3	12	2.1	3	2.0	16	1.6
SAND SHINER	--	--	--	--	--	--	--	--
REDFIN SHINER	--	--	--	--	--	--	--	--
CHANNEL SHINER	--	--	--	--	--	--	--	--
SUCKERMOUTH MINNOW	--	--	18	3.1	6	4.0	24	2.3
BLUNTNOSE MINNOW	2	0.7	21	3.6	20	13.3	43	4.2
CREEK CHUB	42	14.1	39	6.7	9	6.0	90	8.7
RIVER CARPSUCKER	--	--	--	--	--	--	--	--
WHITE SUCKER	13	4.4	--	--	--	--	13	1.3
SPOTTED SUCKER	--	--	--	--	--	--	--	--
SHORTHEAD REDHORSE	--	--	--	--	1	0.7	1	0.1
YELLOW BULLHEAD	21	7.1	10	1.7	5	3.3	36	3.5
PIRATE PERCH	--	--	--	--	1	0.7	1	0.1
WESTERN MOSQUITOFISH	6	2.0	8	1.4	2	1.3	16	1.6
BROOK SILVERSIDE	--	--	--	--	1	0.7	1	0.1
GREEN SUNFISH	13	4.4	8	1.4	36	23.8	57	5.5
BLUEGILL	102	34.2	69	11.8	11	7.3	182	17.6
LONGEAR SUNFISH	7	2.4	12	2.1	8	5.3	27	2.6
REDEAR SUNFISH	--	--	--	--	--	--	--	--
<i>Lepomis</i> HYBRID	--	--	1	0.2	--	--	1	0.1
SPOTTED BASS	1	0.3	--	--	--	--	1	0.1
LARGEMOUTH BASS	--	--	--	--	--	--	--	--
SLOUGH DARTER	1	0.3	--	--	2	1.3	3	0.3
JOHNNY DARTER	4	1.3	4	0.7	11	7.3	19	1.8
<b>Total Fish</b>	<b>298</b>	<b>100.0</b>	<b>583</b>	<b>100.0</b>	<b>151</b>	<b>100.0</b>	<b>1,032</b>	<b>100.0</b>
<b>Total Species</b>	<b>14</b>		<b>12</b>		<b>16</b>		<b>19</b>	

Table 7. Cont.

Survey Period:	19-20 October							
Location:	1		2		3		Combined	
Gear:	EFLONG		EFPRAM		EFPRAM			
Species	No.	%	No.	%	No.	%	No.	%
CENTRAL STONEROLLER	161	35.4	81	15.8	2	0.5	244	17.4
SILVERJAW MINNOW	41	9.0	145	28.3	163	37.3	349	24.8
BIGEYE CHUB	--	--	--	--	2	0.5	2	0.1
EMERALD SHINER	--	--	--	--	29	6.6	29	2.1
STRIPED SHINER	--	--	--	--	2	0.5	2	0.1
SPOTFIN SHINER	5	1.1	8	1.6	30	6.9	43	3.1
SAND SHINER	--	--	--	--	2	0.5	2	0.1
REDFIN SHINER	--	--	--	--	1	0.2	1	0.1
CHANNEL SHINER	--	--	--	--	2	0.5	2	0.1
SUCKERMOUTH MINNOW	--	--	24	4.7	19	4.4	43	3.1
BLUNTNOSE MINNOW	--	--	17	3.3	59	13.5	76	5.4
CREEK CHUB	92	20.2	59	11.5	27	6.2	178	12.7
RIVER CARPSUCKER	--	--	--	--	1	0.2	1	0.1
WHITE SUCKER	9	2.0	--	--	4	0.9	13	0.9
SPOTTED SUCKER	--	--	--	--	4	0.9	4	0.3
SHORTHEAD REDHORSE	--	--	--	--	2	0.5	2	0.1
YELLOW BULLHEAD	4	0.9	4	0.8	2	0.5	10	0.7
PIRATE PERCH	--	--	--	--	3	0.7	3	0.2
WESTERN MOSQUITOFISH	14	3.1	46	9.0	2	0.5	62	4.4
BROOK SILVERSIDE	--	--	--	--	1	0.2	1	0.1
GREEN SUNFISH	13	2.9	14	2.7	32	7.3	59	4.2
BLUEGILL	89	19.6	105	20.5	12	2.8	206	14.7
LONGEAR SUNFISH	6	1.3	3	0.6	5	1.1	14	1.0
REDEAR SUNFISH	--	--	--	--	1	0.2	1	0.1
<i>Lepomis</i> HYBRID	--	--	--	--	--	--	--	--
SPOTTED BASS	--	--	--	--	--	--	--	--
LARGEMOUTH BASS	1	0.2	1	0.2	1	0.2	3	0.2
SLOUGH DARTER	--	--	--	--	1	0.2	1	0.1
JOHNNY DARTER	20	4.4	6	1.2	28	6.4	54	3.8
<b>Total Fish</b>	<b>455</b>	<b>100.0</b>	<b>513</b>	<b>100.0</b>	<b>437</b>	<b>100.0</b>	<b>1,405</b>	<b>100.0</b>
<b>Total Species</b>	<b>12</b>		<b>13</b>		<b>27</b>		<b>27</b>	

**Table 8.** Relative Abundance of Fish Species Collected Seining by Location, September and October 2022.

<b>Survey Period:</b>	<b>21-22 September</b>			<b>19-20 October</b>		
<b>Location:</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>1</b>	<b>2</b>	<b>3</b>
<b>Gear:</b>	<b>SEINE</b>	<b>SEINE</b>	<b>SEINE</b>	<b>SEINE</b>	<b>SEINE</b>	<b>SEINE</b>
<b>Species</b>	<b>Abundance</b>	<b>Abundance</b>	<b>Abundance</b>	<b>Abundance</b>	<b>Abundance</b>	<b>Abundance</b>
CENTRAL STONEROLLER	Common	Common	--	Common	Abundant	--
SILVERJAW MINNOW	~25-30	>30	~60	Common	Abundant	Abundant
BIGEYE CHUB	--	--	--	--	--	4
EMERALD SHINER	--	--	Common	--	--	Common
STRIPED SHINER	--	--	--	--	--	Common
SPOTFIN SHINER	~25	>30	~30	Common	Abundant	Abundant
REDFIN SHINER	--	--	--	--	--	Common
BLUNTNOSE MINNOW	~5	~8	10	Present	Common	Common
CREEK CHUB	~10	--	5	4	Abundant	Common
WHITE SUCKER	Common	Common	--	5	--	--
BLACKSTRIPE TOPMINNOW	3	--	--	--	--	--
WESTERN MOSQUITOFISH	Several	Present	--	Present	Common	--
BROOK SILVERSIDE	--	--	10	--	--	Present
GREEN SUNFISH	--	--	--	--	Present	Present
BLUEGILL	Common	Common	Present	Common	Common	Common
LONGEAR SUNFISH	--	--	1	--	Present	--
LARGEMOUTH BASS	1	1	--	1	Present	1
JOHNNY DARTER	2	2	4	Present	Present	Common
LOGPERCH	1	--	--	--	--	--
<b>Total Species/Location</b>	<b>12</b>	<b>9</b>	<b>9</b>	<b>10</b>	<b>11</b>	<b>13</b>
<b>Total Species</b>	<b>15</b>			<b>17</b>		

**Table 9.** Species Richness and Total Numbers of Fish Collected Electrofishing, September and October 2022.

Parameters	Locations			Total
	1	2	3	
Species Richness				
September	14	12	16	19
October	12	13	27	27
Total Numbers				
September	298	583	151	1,032
October	455	513	437	1,405

**Table 10.** Summary of QHEI Metric Scores in Robinson Creek by Location, September 2022.

<b>Station</b>	<b>Substrate</b>	<b>Cover</b>	<b>Channel</b>	<b>Riparian</b>	<b>Pool/Current</b>	<b>Riffle/Run</b>	<b>Gradient</b>	<b>QHEI Score</b>	<b>Narrative</b>
MPC-1	10.00	13.00	12.50	5.75	10.00	3.50	10.00	<b>64.75</b>	Good
MPC-2	9.50	10.00	12.50	7.50	6.00	2.00	10.00	<b>57.50</b>	Fair
MPC-3	12.50	15.00	10.00	5.00	9.00	3.00	6.00	<b>60.50</b>	Good

**Table 11.** Robinson Creek water quality measurements, September and October 2022 (EF=Electrofishing; SN=Seining).

<u>Station</u>	<u>Date</u>	<u>Time</u>	<u>Temperature (C)</u>	<u>Dissolved Oxygen (ppm)</u>	<u>Specific Conductance (uS/cm)</u>	<u>Secchi (cm)</u>	<u>pH</u>
Location 1-EF	21 September	1335	25.5	9.8	1036	>100	8.4
Location 1-SN	20 September	0930	24.7	6.5	961	--	8.3
Location 2-EF	21 September	0935	29.9	9.6	1767	>78	8.0
Location 2-SN	20 September	1211	30.5	9.6	1636	--	7.9
Location 3-EF	22 September	0902	22.7	7.2	1498	>100	7.9
Location 3-SN	20 September	1545	26.2	9.5	942	--	8.1
Location 1-EF	20 October	1025	8.5	13.1	1386	>100	8.2
Location 1-SN	18 October	750	8.9	11.8	1358	--	8.2
Location 2-EF	19 October	0950	19.6	10.9	1427	>78	7.8
Location 2-SN	18 October	1005	20.1	7.9	1430	--	8.0
Location 3-EF	19 October	1250	11.4	10.5	1252	>100	8.2
Location 3-SN	18 October	1220	11.8	10.0	1285	--	8.1

**Table 12.** Summary of Fish Species Exhibiting DELT Anomalies by Trip and Location, September and October 2022.

**21-22 September**

Anomaly Grouping:	DELT Anomalies						Total With Anomalies	Total Number Examined	Percent Anomalies
	Location:	1		2		3			
Species	No.	%	No.	%	No.	%			
SILVERJAW MINNOW	--	--	12	3.8	--	--	12	356	3.4
SUCKERMOUTH MINNOW	--	--	5	27.8	--	--	5	24	20.8
BLUNTNOSE MINNOW	--	--	--	--	2	10.0	2	43	4.7
YELLOW BULLHEAD	--	--	5	50.0	--	--	5	36	13.9
GREEN SUNFISH	--	--	2	25.0	--	--	2	57	3.5
BLUEGILL	1	1.0	50	72.5	--	--	51	182	28.0
<i>Lepomis</i> HYBRID	--	--	1	100.0	--	--	1	1	100.0
<b>Location Affliction Rates</b>	<b>1</b>	<b>0.3</b>	<b>75</b>	<b>12.9</b>	<b>2</b>	<b>1.3</b>			
<b>Survey Affliction Rates</b>	<b>7.6%</b>						<b>78</b>	<b>1,032</b>	<b>7.6</b>

**19-20 October**

Anomaly Grouping:	DELT Anomalies						Total With Anomalies	Total Number Examined	Percent Anomalies
	Location:	1		2		3			
Species	No.	%	No.	%	No.	%			
SUCKERMOUTH MINNOW	--	--	2	8.3	--	--	2	43	4.7
CREEK CHUB	1	1.1	--	--	--	--	1	178	0.6
YELLOW BULLHEAD	--	--	3	75.0	--	--	3	10	30.0
BLUEGILL	--	--	101	96.2	--	--	101	206	52.4
LONGEAR SUNFISH	1	16.7	--	--	--	--	1	14	7.1
<b>Location Affliction Rates</b>	<b>2</b>	<b>0.4</b>	<b>106</b>	<b>20.7</b>	<b>--</b>	<b>--</b>			
<b>Survey Affliction Rates</b>	<b>7.7%</b>						<b>108</b>	<b>1,405</b>	<b>7.7</b>



**Table 13.** Summary of DELT Anomalies by Trip and Location, September and October 2022.

Survey Period:		21-22 September				19-20 October			
Location:		1	2	3	Combined	1	2	3	Combined
Species	Anomaly	No.	No.	No.	No.	No.	No.	No.	No.
SILVERJAW MINNOW	Erosion	--	12	--	12	--	--	--	--
SPOTFIN SHINER									
SUCKERMOUTH MINNOW	Deformity	--	1	--	1	--	--	--	--
	Erosion	--	4	--	4	--	2	--	2
BLUNTNOSE MINNOW	Deformity	--	--	1	1	--	--	--	--
	Erosion	--	--	1	1	--	--	--	--
	Lesions-Ulcers	--	--	1	1	--	--	--	--
CREEK CHUB	Deformity	--	--	--	--	1	--	--	1
YELLOW BULLHEAD	Erosion	--	5	--	5	--	3	--	3
GREEN SUNFISH	Erosion	--	2	--	2	--	--	--	--
BLUEGILL	Erosion	1	50	--	51	--	101	--	101
LONGEAR SUNFISH	Deformity	--	--	--	--	1	--	--	1
<i>Lepomis</i> HYBRID	Erosion	--	1	--	1	--	--	--	--

**Table 14.** ANOVA (Type III) reporting comparisons of weight, length, and CF across water treatments for Fathead Minnow after 30 and 60 days of exposure, controlling for variation by temperature.

<b>Weight (Day 30)</b>	SS	df	<i>F</i>	<i>p</i>
(Intercept)	1279.361	1	4386.991	<0.001
Temperature	7.846	1	26.905	<0.001
Treatment	2.982	3	3.409	0.02
Temp:Treatment	1.585	3	1.812	0.149
Residuals	31.496	108		

<b>Length (Day 30)</b>	SS	df	<i>F</i>	<i>p</i>
(Intercept)	488938.948	1	27114.485	<0.001
Temperature	567.280	1	31.459	<0.001
Treatment	28.936	3	0.535	0.659
Temp:Treatment	71.667	3	1.325	0.27
Residuals	2001.595	111		

<b>Weight (Day 60)</b>	SS	df	<i>F</i>	<i>p</i>
(Intercept)	1552.168	1	5123.302	<0.001
Temperature	12.952	1	42.753	<0.001
Treatment	8.586	3	9.446	<0.001
Temp:Treatment	0.679	3	0.747	0.526
Residuals	33.326	110		

<b>Length (Day 60)</b>	SS	df	<i>F</i>	<i>p</i>
(Intercept)	531166.107	1	38846.317	<0.001
Temperature	485.091	1	35.477	<0.001
Treatment	217.767	3	5.309	0.002
Temp:Treatment	61.677	3	1.504	0.218
Residuals	1490.414	109		

<b>CF (Day 60)</b>	SS	df	<i>F</i>	<i>p</i>
(Intercept)	156.325	1	15177.525	<0.001
Temperature	0.001	1	0.139	0.71
Treatment	0.068	3	2.205	0.092
Temp:Treatment	0.034	3	1.105	0.35
Residuals	1.112	108		

**Table 15.** Welch's test reporting comparison of CF across water treatments and temperature for Fathead Minnow after 30 days of exposure.

<b>CF (Day 30, Water Treatment)</b>				<b>CF (Day 30, Temperature)</b>			
Num. df	Denom. df	F	p	Num. df	Denom. df	F	p
3.000	60.817	4.395	0.007	1.000	98.289	15.085	<0.001

**Table 16.** Results of post-hoc Dunnett comparisons of weight, length, and CF across water treatments for Fathead Minnow after 30 and 60 days of exposure.

<b>Weight (Day 30)</b>				
Comparison	Estimates	SE	t value	p
Upstream - Control	-0.242	0.158	-1.536	0.294
Effluent - Control	-0.438	0.161	-2.726	0.021
Downstream - Control	-0.147	0.156	-0.942	0.669

<b>Weight (Day 60)</b>				
Comparison	Estimates	SE	t value	p
Upstream - Control	-0.084	0.167	-0.505	0.922
Effluent - Control	-0.663	0.169	-3.936	<0.001
Downstream - Control	-0.005	0.167	-0.028	1

<b>CF (Day 30)</b>				
Comparison	Estimates	SE	t value	p
Upstream - Control	0.020	0.035	0.560	0.899
Effluent - Control	-0.084	0.035	-2.359	0.053
Downstream - Control	-0.060	0.036	-1.677	0.228

<b>Length (Day 60)</b>				
Comparison	Estimates	SE	t value	p
Upstream - Control	0.129	1.117	0.115	0.999
Effluent - Control	-2.533	1.097	-2.308	0.06
Downstream - Control	1.234	1.107	1.115	0.548

<b>CF (Day 60)</b>				
Comparison	Estimates	SE	t value	p
Upstream - Control	-0.039	0.027	-1.466	0.331
Effluent - Control	-0.064	0.026	-2.451	0.042
Downstream - Control	-0.050	0.027	-1.898	0.15

**Table 17.** ANOVA (Type III) reporting comparison of cortisol concentrations (pg/g) across water treatments for Fathead Minnow fins after 30 and 60 days of exposure, controlling for variation by temperature.

<b>Cortisol (Day 30)</b>					<b>Cortisol (Day 60)</b>				
	SS	df	<i>F</i>	<i>p</i>		SS	df	<i>F</i>	<i>p</i>
(Intercept)	193684862	1	39.526	<0.001	(Intercept)	276381654.0	1	118.099	<0.001
Temperature	31130048	1	6.353	0.02	Temperature	884882.2	1	0.378	0.546
Treatment	5023289	3	0.342	0.795	Treatment	36631771.2	3	5.218	0.008
Temp:Treatment	4402430	3	0.299	0.825	Temp:Treatment	18466488.1	3	2.630	0.08
Residuals	102903605	21			Residuals	44464805.7	19		

**Table 18.** Results of post-hoc Dunnett comparisons of cortisol across water treatments for Fathead Minnow fins after 60 days of exposure, at 20°C.

<b>Cortisol (Day 60, 20 Degrees Celsius)</b>				
Comparison	Estimates	SE	t value	p
Upstream - Control	344.563	1136.591	0.303	0.982
Effluent - Control	4465.181	1227.659	3.637	0.012
Downstream - Control	948.434	1227.659	0.773	0.792

**Table 19.** Differential white blood cell counts in Fathead Minnow bioassay.

<b>Treatment</b>	<b>Temperature (°C)</b>	<b>Lymphocytes (%)</b>	<b>Monocytes (%)</b>	<b>Granulocytes (%)</b>
CON	20	96.9	2.3	0.8
CON	30	96.8	2.3	0.9
DNS	20	96.7	2.6	0.7
DNS	30	97.2	2.1	0.7
EFF	20	97.0	2.1	0.9
EFF	30	97.1	2.2	0.8
UPS	20	96.5	2.5	1.0
UPS	30	96.8	2.3	0.9

**Table 20.** ANOVA (Type III) reporting comparison of lipid content (%) across water treatments for Fathead Minnow livers after 30 and 60 days of exposure, controlling for variation by temperature.

<b>Lipid Content (Day 30)</b>					<b>Lipid Content (Day 60)</b>				
	SS	df	<i>F</i>	<i>p</i>		SS	df	<i>F</i>	<i>p</i>
(Intercept)	464.404	1	296.604	<0.001	(Intercept)	545.303	1	140.330	<0.001
Temperature	8.470	1	5.409	0.028	Temperature	34.083	1	8.771	0.006
Treatment	13.706	3	2.918	0.052	Treatment	10.415	3	0.893	0.456
Temp:Treatment	19.490	3	4.149	0.015	Temp:Treatment	6.050	3	0.519	0.672
Residuals	42.275	27			Residuals	120.462	31		



**Table 21.** Results of post-hoc Dunnett comparisons of lipid content (%) across water treatments for Fathead Minnow fins after 30 days of exposure, separated by temperature.

<b>Lipid Content (Day 30, 20 Degrees Celsius)</b>					<b>Lipid Content (Day 30, 30 Degrees Celsius)</b>				
Comparison	Estimates	SE	t value	p	Comparison	Estimates	SE	t value	p
Upstream - Control	-0.777	0.807	-0.963	0.646	Upstream - Control	1.094	0.957	1.144	0.525
Effluent - Control	1.448	0.807	1.795	0.208	Effluent - Control	-0.369	1.070	-0.345	0.968
Downstream - Control	-0.381	0.807	-0.472	0.929	Downstream - Control	-1.821	0.957	-1.903	0.18

**Table 22.** ANOVA (Type III) reporting comparison of mean relative mRNA of *cat*, *sod*, *cyp1a*, and *gst* across water treatments for Fathead Minnow livers after 30 and 60 days of exposure, controlling for variation by temperature. ^^ = indicates a model excluding an influential data point on the basis of Cook's distance.

<i>cat</i> (Day 30)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	0.450	1	2.174	0.152
Temperature	0.308	1	1.488	0.233
Treatment	1.166	3	1.876	0.157
Temp:Treatment	0.346	3	0.556	0.648
Residuals	5.800	28		

<i>cat</i> (Day 60)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	0.553	1	5.491	0.026
Temperature	0.409	1	4.064	0.053
Treatment	0.737	3	2.438	0.085
Temp:Treatment	0.238	3	0.787	0.511
Residuals	2.820	28		

<i>gst</i> (Day 30)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	2.452	1	4.510	0.042
Temperature	3.002	1	5.522	0.026
Treatment	1.058	3	0.649	0.590
Temp:Treatment	3.116	3	1.910	0.149
Residuals	16.308	30		

<i>gst</i> (Day 60)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	3.753	1	5.804	0.023
Temperature	7.346	1	11.361	0.002
Treatment	0.881	3	0.454	0.717
Temp:Treatment	1.757	3	0.906	0.451
Residuals	18.104	28		

<i>cyp1a</i> <sup>^^</sup> (Day 30)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	3.278	1	5.638	0.025
Temperature	1.220	1	2.098	0.159
Treatment	3.857	3	2.211	0.109
Temp:Treatment	0.593	3	0.340	0.797
Residuals	16.279	28		

<i>cyp1a</i> (Day 60)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	0.535	1	1.493	0.232
Temperature	0.000	1	0.001	0.971
Treatment	0.628	3	0.584	0.630
Temp:Treatment	0.453	3	0.422	0.739
Residuals	10.388	29		

<i>sod</i> (Day 30)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	1.810	1	0.654	0.427
Temperature	0.015	1	0.005	0.942
Treatment	22.292	3	2.684	0.070
Temp:Treatment	18.362	3	2.211	0.114
Residuals	63.672	23		

<i>sod</i> (Day 60)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	0.080	1	0.053	0.820
Treatment	12.939	3	2.863	0.063
Residuals	30.132	20		

**Table 23.** Results of post-hoc Dunnett comparisons of Ln(Fold Change) for Fathead Minnow livers across water treatments.  $\hat{\hat{}}$  = indicates a model excluding an influential data point on the basis of Cook's distance.

<i>cyp1a</i> $\hat{\hat{}}$ (Day 30)					<i>cat</i> (Day 60)				
Comparison	Estimates	SE	t value	p	Comparison	Estimates	SE	t value	p
Upstream - Control	0.600	0.288	2.082	0.119	Upstream - Control	-0.156	0.213	-0.732	0.809
Effluent - Control	1.303	0.265	4.922	<0.001	Effluent - Control	-0.480	0.201	-2.392	0.061
Downstream - Control	0.696	0.250	2.790	0.026	Downstream - Control	-0.411	0.201	-2.048	0.124

<i>sod</i> (Day 30)				
Comparison	Estimates	SE	t value	p
Upstream - Control	0.423	1.355	0.312	0.977
Effluent - Control	-0.322	1.212	-0.266	0.986
Downstream - Control	-2.713	1.267	-2.140	0.103

**Table 24.** ANOVA (Type III) and Welch's test reporting comparisons of weight, length, and CF across Robinson Creek field sites for Spottfin Shiner sampled in September and October 2022.

<b>Weight (Sept 2022)</b>				<b>Weight (Oct 2022)</b>			
Num. df	Denom. df	F	p	Num. df	Denom. df	F	p
2.000	36.628	2.698	0.081	2.000	33.252	19.740	<0.001

<b>Length (Sept 2022)</b>					<b>Length (Oct 2022)</b>				
	SS	df	F	p		SS	df	F	p
(Intercept)	345932.508	1	3171.823	<0.001	(Intercept)	348638.346	1	3215.994	<0.001
Site	421.092	2	1.930	0.152	Site	2415.833	2	11.142	<0.001
Residuals	8288.882	76			Residuals	8022.167	74		

<b>CF (Sept 2022)</b>				<b>CF (Oct 2022)</b>			
Num. df	Denom. df	F	p	Num. df	Denom. df	F	p
2.000	44.602	11.843	<0.001	2.000	38.006	35.789	<0.001

**Table 25.** Results of post-hoc Dunnett comparisons of weight, length, and CF across Robinson Creek field sites for Spottfin Shiner sampled in September and October 2022.

**Weight (Sept 2022)**

Comparison	Estimates	SE	t value	p
LOC 2 - LOC 1	-0.728	0.324	-2.243	0.05
LOC 3 - LOC 1	-0.991	0.329	-3.013	0.007

**CF (Sept 2022)**

Comparison	Estimates	SE	t value	p
LOC 2 - LOC 1	0.040	0.019	2.100	0.068
LOC 3 - LOC 1	-0.044	0.019	-2.375	0.036

**Weight (Oct 2022)**

Comparison	Estimates	SE	t value	p
LOC 2 - LOC 1	0.387	0.402	0.963	0.508
LOC 3 - LOC 1	-1.428	0.402	-3.556	0.001

**Length (Oct 2022)**

Comparison	Estimates	SE	t value	p
LOC 2 - LOC 1	-0.833	3.161	-0.264	0.945
LOC 3 - LOC 1	-12.000	3.161	-3.797	0.001

**CF (Oct 2022)**

Comparison	Estimates	SE	t value	p
LOC 2 - LOC 1	0.124	0.018	6.770	<0.001
LOC 3 - LOC 1	-0.011	0.018	-0.609	0.748

**Table 26.** ANOVA (Type III) reporting comparison of cortisol concentrations (pg/g) across Robinson Creek field sites for Spottfin Shiner fins sampled in September and October 2022.

<b>Cortisol (Sept 2022)</b>					<b>Cortisol (Oct 2022)</b>				
	SS	df	<i>F</i>	<i>p</i>		SS	df	<i>F</i>	<i>p</i>
(Intercept)	2095852952	1	75.456	<0.001	(Intercept)	3501451029	1	29.396	<0.001
Site	145980114	2	2.628	0.097	Site	478655814	2	2.009	0.163
Residuals	555517768	20			Residuals	2144060745	18		

**Table 27.** Differential white blood cell counts in Spotfin Shiner from Robinson Creek.

<b>Site</b>	<b>Temperature (°C)</b>	<b>Lymphocytes (%)</b>	<b>Monocytes (%)</b>
Location 1	97.5	1.7	0.8
Location 2	97.0	2.3	0.8
Location 3	96.9	2.2	0.9

**Table 28.** ANOVA (Type III) and Welch's test reporting comparison of lipid content (%) across Robinson Creek field sites for Spotfin Shiner livers sampled in September and October 2022.

<b>Lipid Content (Sept 2022)</b>					<b>Lipid Content (Oct 2022)</b>			
	SS	df	<i>F</i>	<i>p</i>	Num. df	Denom. df	<i>F</i>	<i>p</i>
(Intercept)	140.053	1	353.382	<0.001	2.000	15.223	4.658	0.03
Site	6.937	2	8.752	0.001				
Residuals	9.908	25						



**Table 29.** Results of post-hoc Dunnett comparison of lipid content (%) across Robinson Creek field sites for Spotfin Shiner livers.

<b>Lipid Content (Sept 2022)</b>					<b>Lipid Content (Oct 2022)</b>				
Comparison	Estimates	SE	t value	p	Comparison	Estimates	SE	t value	p
LOC 2 - LOC 1	0.466	0.282	1.655	0.192	LOC 2 - LOC 1	1.199	0.384	3.124	0.009
LOC 3 - LOC 1	-0.779	0.299	-2.608	0.028	LOC 3 - LOC 1	-0.081	0.406	-0.200	0.971

**Table 30.** ANOVA (Type III) reporting comparison of mean relative mRNA of *cat*, *sod*, *cyp1a*, and *gst* across Robinson Creek field sites for Spottfin Shiner livers sampled in September and October 2022.

<i>cat</i> (Sept 2022)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	30.990	1	21.547	0.000
Site	0.603	2	0.210	0.812
Residuals	34.517	24		

<i>cat</i> (Oct 2022)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	0.001	1	0.003	0.960
Site	0.497	2	0.724	0.497
Residuals	6.868	20		

<i>gst</i> (Sept 2022)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	43.416	1	21.939	0.00
Site	1.998	2	0.505	0.61
Residuals	43.537	22		

<i>gst</i> (Oct 2022)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	1.905	1	5.829	0.026
Site	2.959	2	4.527	0.025
Residuals	6.209	19		

<i>cyp1a</i> (Sept 2022)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	45.640	1	20.392	0.000
Site	3.531	2	0.789	0.466
Residuals	51.477	23		

<i>cyp1a</i> (Oct 2022)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	0.031	1	0.049	0.828
Site	1.443	2	1.147	0.340
Residuals	11.319	18		

<i>sod</i> (Sept 2022)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	60.929	1	27.488	0.000
Site	2.858	2	0.645	0.534
Residuals	48.765	22		

<i>sod</i> (Oct 2022)	SS	df	<i>F</i>	<i>p</i>
(Intercept)	0.690	1	1.918	0.181
Site	1.138	2	1.581	0.229
Residuals	7.560	21		

**Table 31.** Results of post-host Dunnett comparisons of Ln (Fold Change) for *gst* of October 2022 Spotfin Shiner livers across field sites.

<b><i>gst</i> (Oct 2022)</b>				
Comparison	Estimates	SE	t value	p
LOC 2 - LOC 1	0.817	0.296	2.760	0.023
LOC 3 - LOC 1	0.067	0.296	0.228	0.964

## **Appendix A**

# **DELTs Fish Community, Bioassay, and Fish Health Assessment Study Plan**

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# **Study Plan for the Assessment of Deformity, Erosion, Lesion, and Tumor (DELT) Anomalies at Marathon Petroleum Company's Robinson Refinery**

*Prepared for*

Marathon Petroleum Company, LP  
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August 2022  
Version: FINAL  
EA Project No. 1604001

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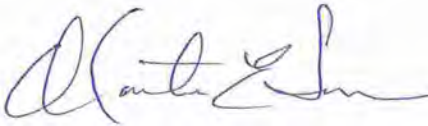
# **Study Plan for the Assessment of Deformity, Erosion, Lesion, and Tumor (DELT) Anomalies at Marathon Petroleum Company's Robinson Refinery**

*Prepared for*

Marathon Petroleum Company LP  
400 S Marathon Avenue  
Robinson, IL 62454

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Deerfield, IL 60015

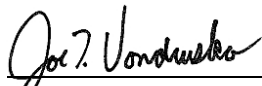


10 August 2022

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Marty Sneen  
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Date



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10 August 2022

Date

August 2022  
Version: FINAL  
EA Project No. 1604001



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Appendix D. Illinois Scientific Collection Permits

Appendix E. Field Sampling Data Sheet

Appendix F. Multiprobe Water Quality Monitoring Instruments

Appendix G. MPC Safety Procedure #12 GENERAL SAFETY RULES

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**LIST OF ACRONYMS AND ABBREVIATIONS**

°F	Degrees Fahrenheit
°C	Degrees Celsius
ANZI	American National Standards Institute
ASTM	American Society for Testing and Materials
ATHEL	Alternative Thermal Effluent Limitation
cat	Catalase
cDNA	Complimentary DNA
COVID-19	Coronavirus Disease 2019
CPE	Catch-Per-Unit-Effort
DC	Direct Current
DELT	Deformities, Erosion, Lesions/Ulcers, and Tumors
DISA	Defense Information Systems Agency
DNA	Deoxyribonucleic Acid
DO	Dissolved Oxygen
EA	EA Engineering, Science, and Technology, Inc., PBC
EDD	Electronic Data Deliverable
ELISA	Enzyme-Linked Immunosorbent Assay
EPA	Environmental Protection Agency
FDA	U.S. Food and Drug Administration
FHM	Fathead Minnow <i>Pimephales promelas</i>
FHI	Fish Health Index
FR	Fire Retardant
ft.	Feet
g	Gram(s)
g/L	Grams per Liter
GADPH	Glyceraldehyde 3 Phosphate Dehydrogenase
GPS	Global Positioning System
gst	Glutathione-s-Transferase
hr.	Hour(s)
lbs.	Pound(s)
ID	Identification
IDNR	Illinois Department of Natural Resources
IPCB	Illinois Pollution Control Board
MBI	Midwest Biodiversity Institute
mg/L	Milligrams per Liter

mL	Milliliters
mm	Millimeters
MPC	Marathon Petroleum Corporation
NBS	National Bureau of Standards
NFPA	National Fire Protection Board
oz.	Ounce(s)
PAH	Polynuclear Aromatic Hydrocarbons
PBI	Polybenzimidazole
pH	Potential Hydrogen
POTW	Publicly Owned Treatment Works
PPE	Personal Protective Equipment
QA/QC	Quality Assurance/Quality Control
qPCR	Quantitative Polymerase Chain Reaction
RNA	Ribonucleic Acid
RM	River Mile
sod	Superoxide Dismutase
TWIC	Transportation Worker Identification Card
µg/dL	Micogram per Deciliter
µL	Microliter
µS/cm	Microsiemens per centimeter
USEPA	United States Environmental Protection Agency
uv	Ultraviolet
vol	Volume
WBC	White Blood Cells

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## 1. INTRODUCTION

As set forth in Order and Opinion PBC 18-49 and in response to comments provided by the Illinois Department of Natural Resources (IDNR), the Illinois Pollution Control Board (IPCB) found that the record did not contain adequate information to determine if the synergistic effect of Marathon Petroleum Company's (MPC) Robinson Refinery thermal discharge and non-thermal stressors in Robinson Creek is causing an increased incidence of deformity, erosion, lesion, and tumor (DELT) anomalies on fish. Given that the proposed alternative thermal effluent limitations (ATELs) include a mixing zone without a zone of passage, the IPCB required as a condition to the ATELs that MPC conduct a study as suggested by the IDNR (PBC 18-49, 7 July 2020 IDNR Response, Attachment C). This study was designed to follow the IDNR recommended study with modifications to accommodate field implementation.

DELT anomalies are the group of anomalies for which a clear relationship has been established between their incidence (percentage) and water quality (Ohio EPA 1987). A high frequency of DELT anomalies is a good indication of a stress caused by sublethal stresses, intermittent stresses, and chemically contaminated substrates. The following is an overview of DELT anomalies and their causes in freshwater fishes:

- Deformities – These anomalies can include malformation of the head, spinal vertebrae, fins, barbels, and abdomen, and have a variety of causes including, but not limited to, toxic chemicals, heavy metals, viral and bacterial (e.g., *Mycobacterium*) infections, and parasites (e.g., *Myxobolus cerebralis*; Post 1983) (Ohio EPA 2015).
- Eroded fin, gill cover, barbel, or other body part – These are the result of chronic disease caused principally by flexibacteria invading the tissue and causing necrosis (Post 1983). Necrosis of the fins may also be caused by gryodactylids, a small trematode parasite (Ohio EPA 2015).
- Lesions and Ulcers – These appear as open sores or exposed tissue and can be caused by viral (e.g., *Lymphocystis*) and bacterial (e.g., *Flexibacter columnaris*, *Aeromonas*, *Vibrio*) infections (Ohio EPA 2015).
- Tumors – These result from the loss of carefully regulated cellular proliferative growth in tissue and are generally referred to as neoplasia (Post 1983). In wild fish populations, tumors can be the result of exposure to toxic chemicals. Baumann et al. (1987) identified polynuclear aromatic hydrocarbons (PAHs) as the cause of hepatic tumors in Brown Bullhead from the Black River (Ohio). Viral infections (e.g., *Lymphocystis*) can also cause tumors. Parasites (e.g., *Glugea anomala* and *Ceratonova shasta*; Post 1983) may cause tumor-like masses, but these are not counted as tumors. Parasite masses can be squeezed and broken between the thumb and forefinger, whereas true tumors are firm and not easily broken (Ohio EPA 2015).

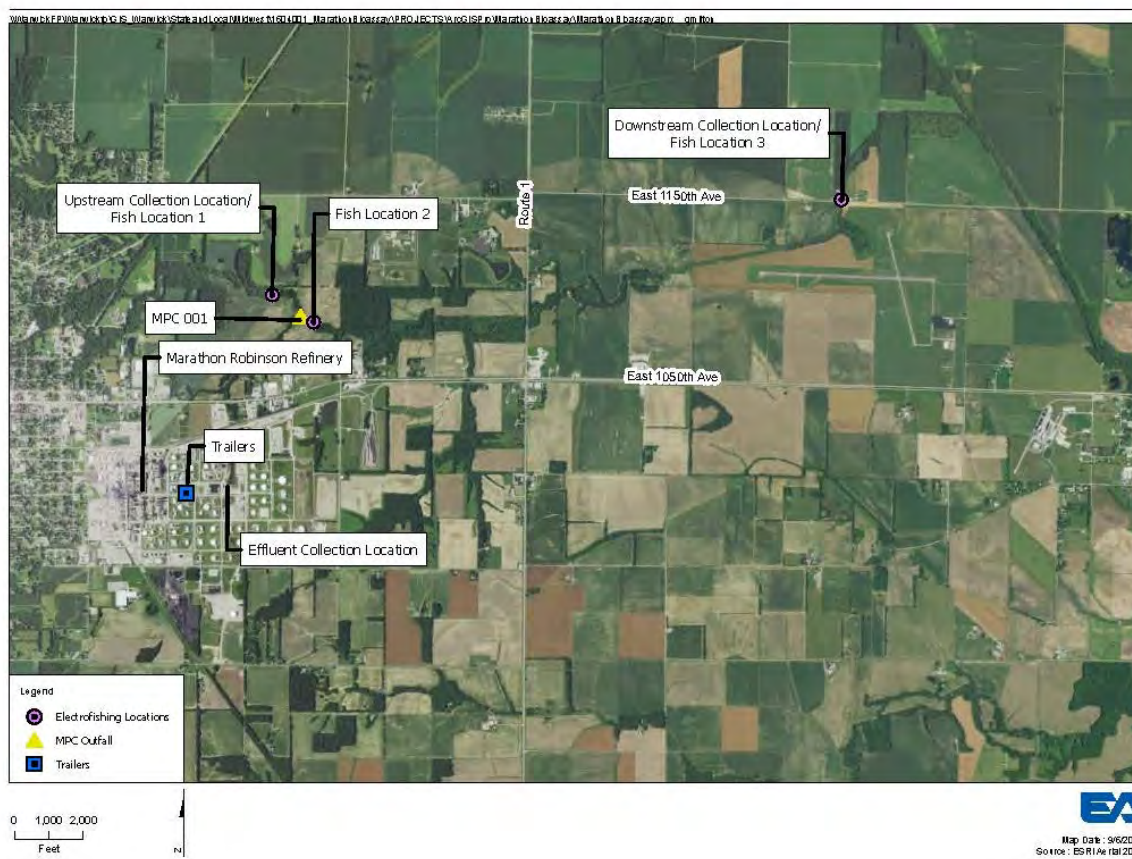
This study consists of three elements: onsite thermal bioassay, field collections and DELTs assessment, and fish health assessment. The primary objective of this study was to determine whether the Robinson Refinery thermal discharge is causing an increased incidence of DELTs on

fish in Robinson Creek, particularly in Bigeye Chub and similar species. We hypothesized that Fathead Minnow exposed to the refinery effluent and Spottfin Shiner collected closest to the refinery effluent outflow would respond with decreased growth and lipid reserves; increased prevalence of DELTs and increased cortisol levels; altered blood cells differentials in fish showing DELT anomalies; and with a dysregulation in the expression of detoxification and oxidative stress genes compared to controls. We also hypothesized that increased water temperatures would exacerbate these changes and impact survival.

## 2. METHODS AND MATERIALS

### 2.1 ONSITE THERMAL BIOASSAY

The on-site thermal bioassay will consist of three site exposures and one control. The site exposures will consist of upstream, effluent, and downstream waters (Figure 1) conducted simultaneously with a dechlorinated tap water exposure (the control). The testing will be conducted at two temperatures to evaluate thermal stress. The studies will be conducted for 60 days or as long as the refinery remains within normal operating conditions. Study organisms will consist of adult (~6-month-old) male Fathead Minnows (*Pimephales promelas*). The two temperatures will represent a background cold water stream maximum condition and an elevated temperature condition that will mimic summer/fall variations.



**Figure 1. Aerial View of the Upstream, Effluent, and Downstream Exposure Water Collection Points**

USEPA states that when choosing test organisms, one should select a species that is representative of resident organisms, sensitive to site contaminants, relevant to the overall assessment endpoints, and consistent with data quality objectives (US EPA 1992). The test organisms should serve as surrogates for organisms present on the site. Based on this broadly

accepted framework, a commercially available minnow species, Fathead Minnow, which is in the same genus as Bluntnose Minnow (*Pimephales notatus*) will be used. Approximately six-month-old, sexually mature, male Fathead Minnows will be obtained from a scientific organism vendor (Aquatic BioSystems, Fort Collins, Colorado). Aquatic BioSystems is a full-service organism culturing facility specializing in the production and distribution of freshwater and marine organisms for aquatic toxicology, biomonitoring and other research activities. The organisms are completely laboratory reared using the latest information and technology available. This ensures the consistent production of organisms that are of the highest quality.

Test vessels will be of sufficient volume to not exceed the organism loading requirements. Loading will not exceed 7 grams/liter (g/L) in any chamber at test temperatures of 15°C and below. At 25°C, loading will not exceed 2.5 g/L at any time. In order to ensure applicable loading rates, testing will be conducted in 50-gallon plastic barrel troughs. Additionally, a 300-gallon reservoir of water will be recirculated through the testing chambers to increase the total water volume and decrease the total concentration of waste products. This water will be refreshed every other day. Each trough will contain 25-30 fish per tank. Three replicate tanks will be used for each test water. Fish will be randomly assigned to each test container.

The test vessels will be set-up in two separate trailers (Figures 2 and 3) with the same treatments (Control, Upstream, Effluent, and Downstream) but at different temperatures. In one environmentally controlled trailer, test vessels will be maintained at 20°C ±2°C while test vessels in the second environmentally controlled trailer will be maintained at 30°C ±2°C. Both trailers will have 16-hour light and 8-hour dark photoperiods with room temperatures monitored continuously.



**Figure 2. Aerial View of Routes to the Bioassay Trailers**



**Figure 3. Bioassay Trailers Footprint adjacent to the Wastewater Filter Press Building**

Water quality (temperature, pH, dissolved oxygen and conductivity) of the test solutions will be measured daily in one replicate per test concentration using a Star Orion A329 multimeter. Additionally, select parameters will be measured at three times throughout the study (i.e., beginning, approximate midpoint, and end). Based on the duration of testing, previous water chemistry results from the 316(a) demonstration (MBI 2017), and constituents commonly associated with DELTs (OEPA 1987), the following parameters were selected for analysis:

**Table 1. Water Chemistry Analytes to be Measured during the Thermal Bioassay Study**

1. Acenaphthene	44. Methylene chloride	87. Dieldrin
2. Acrolein	45. Bromoform	88. Chlordane
3. Acrylonitrile	46. Bromomethane	89. 4,4-DDT
4. Benzene	47. Chloromethane	90. 4,4-DDE
5. Benzidine	48. Dichlorobromomethane	91. 4,4-DDD
6. Carbon tetrachloride	49. Bromodichloromethane	92. Alpha-endosulfan
7. Chlorobenzene	50. Hexachloro-1,3,-butadiene	93. Beta-endosulfan
8. 1,2,4-trichlorobenzene	51. Hexachlorocyclopentadiene	94. Endosulfan sulfate
9. Hexachlorobenzene	52. Isophorone	95. Endrin
10. 1,2-dichloroethane	53. Naphthalene	96. Endrin aldehyde
11. 1,1,1-trichloroethane	54. Nitrobenzene	97. Heptachlor
12. Hexachloroethane	55. 2-nitrophenol	98. Heptachlor epoxide
13. 1,1-dichloroethane	56. 4-nitrophenol	99. Alpha-BHC
14. 1,1,2-trichloroethane	57. 2,4-dinitrophenol	100. Beta-BHC
15. 1,1,2,2-tetrachloroethane	58. N-nitrosodimethylamine	101. Gamma-BHC
16. Chloroethane	59. N-nitrosodiphenylamine	102. Delta-BHC
17. Bis(2-chloroethyl) ether	60. N-nitrosodi-n-propylamine	103. PCB-1242 (Arochlor 1242)



18. 2-chloroethyl vinyl ethers	61. Pentachlorophenol	104. PCB-1254 (Arochlor 1254)
19. 2-chloronaphthalene	62. Phenol	105. PCB-1221 (Arochlor 1221)
20. 2,4,6-trichlorophenol	63. Bis(2-ethylhexyl) phthalate	106. PCB-1232 (Arochlor 1232)
21. Chloroform	64. Butyl benzyl phthalate	107. PCB-1248 (Arochlor 1248)
22. 2-chlorophenol	65. Di-n-Butyl Phthalate	108. PCB-1260 (Arochlor 1260)
23. 1,2-dichlorobenzene	66. Di-n-octyl phthalate	109. PCB-1016 (Arochlor 1016)
24. 1,3-dichlorobenzene	67. Diethyl Phthalate	110. Toxaphene
25. 1,4-dichlorobenzene	68. Dimethyl phthalate	111. Antimony
26. 3,3-dichlorobenzidine	69. Benzo(a) anthracene	112. Arsenic
27. 1,1-dichloroethylene	70. Benzo(a) pyrene	
28. 1,2-trans-dichloroethylene	71. Benzo(b) fluoranthene	113. Beryllium
29. 2,4-dichlorophenol	72. Benzo(k) fluoranthene	114. Cadmium
30. 1,2-dichloropropane	73. Chrysene	115. Chromium
31. 1,3-cis-dichloropropene	74. Acenaphthylene	116. Copper
32. 1,3-trans-dichloropropene	75. Anthracene	117. Cyanide, Total
33. 2,4-dimethylphenol	76. Benzo(ghi) perylene	118. Lead
34. 4,6-dinitro-2-methylphenol	77. Fluorene	119. Mercury
35. 2,4-dinitrotoluene	78. Phenanthrene	120. Nickel
36. 2,6-dinitrotoluene	79. Dibenzo(a,h) anthracene	121. Selenium
37. 1,2-diphenylhydrazine	80. Indeno (1,2,3-cd) pyrene	122. Silver
38. Ethylbenzene	81. Pyrene	123. Thallium
39. Fluoranthene	82. Tetrachloroethylene	124. Zinc
40. 4-chlorophenyl phenyl ether	83. Toluene	125. 2,3,7,8-TCDD
41. 4-bromophenyl phenyl ether	84. Trichloroethylene	
42. Bis(2-chloroisopropyl) ether	85. Vinyl chloride	
43. Bis(2-chloroethoxy) methane	86. Aldrin	

For each of the three collection periods, eight (8) water samples (4 exposure waters x 2 temperatures) will be collected and analyzed for the aforementioned parameters. MPC will be responsible for contracting the laboratory, which includes obtaining the required sample bottles prior to each collection and shipment of collected samples to the laboratory. EA will be responsible for collecting the samples and documenting them on a chain-of-custody form. General guidance for Surface Water Sampling, Sample Preservation and Container Requirements, and Chain-of-Custody Forms are provided in Appendices A, B, and C, respectively. Sample identification nomenclature is provided in Table 2:

**Table 2. Water Chemistry Sample Identification Nomenclature**

Exposure Temperature	Exposure Water	Sample Identification
20°C	Control (CON)	20-CON-ddMMMyyyy <sup>(a)</sup>
20°C	Upstream (UPS)	20-UPS-ddMMMyyyy
20°C	Effluent (EFF)	20-EFF-ddMMMyyyy
20°C	Downstream (DNS)	20-DNS-ddMMMyyyy
30°C	Control (CON)	30-CON-ddMMMyyyy
30°C	Upstream (UPS)	30-UPS-ddMMMyyyy
30°C	Effluent (EFF)	30-EFF-ddMMMyyyy
30°C	Downstream (DNS)	30-DNS-ddMMMyyyy

(a) For example, 25JUL2022

Each test day, test organisms will be visually observed to record any mortalities and the presence or absence of DELT anomalies (Ohio EPA 2015). Dead organisms will be removed when observed and also examined for DELTs. If lesions are present, they will be swabbed and submitted for bacterial analysis.

Water for each of the three treatments (effluent, upstream, and downstream) and control will be exchanged every other day. Water from each of the locations will be collected using a dedicated stainless steel submersible pump. The water will be pumped into a 275-gallon plastic tote and transported to the testing trailer. The water will be allowed to acclimate to the test temperature for at least 1 hour prior to use. The water will be pumped from the tote to the reservoir tank in the testing trailers. Prior to the transfer, the reservoir tanks and accumulated waste from the exposure tanks will be pumped out to the facilities holding basin.

The system will be set-up as a modified flow through system, whereby the water will be recirculated through the tanks at a rate of approximately two volume addition per day. Flow rate will be documented weekly. The flow through system for each water source (i.e., Control, Upstream, Effluent, etc.) will be replenished every other day to minimize loss in water volume and water quality due to degradation, uptake, or evaporation.

In addition to the physical observations, fish health and stress will be assessed at beginning, midpoint, and end of the study. Details of the field and laboratory analysis for fish stress and health markers as well as lesion bacterial sample collection are provided in Section 2.3 and will be processed and analyzed in partnership with Dr. Maria Soledad (Marisol) Sepúlveda's laboratory at Purdue University.

## 2.2 FIELD COLLECTIONS IN ROBINSON CREEK

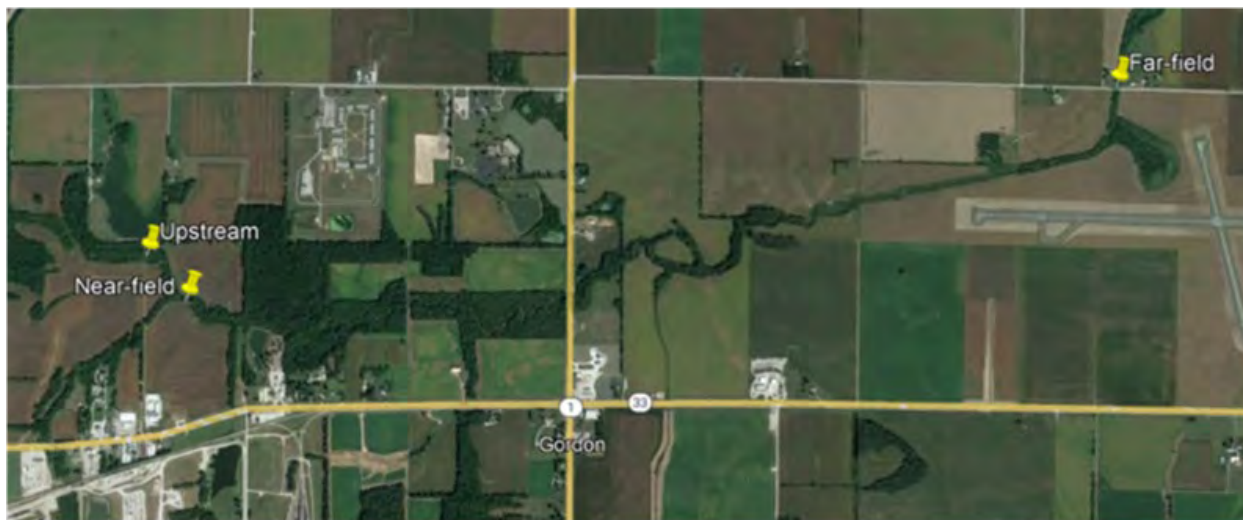
To evaluate fish health along a gradient both upstream and downstream of the discharge, three sampling zones will be established (Table 3 and Figure 4).

**Table 3. Descriptions of Robinson Creek Sampling Zones**

Zone	RM	Description
1	5.2	Ambient conditions, upstream of the MPC thermal discharge and downstream of the Robinson Publicly Owned Treatment Works (POTW). This zone will be near MBI's Location RC04 (MBI 2017).
2	5.0	Near-field zone beginning immediately downstream of the MPC thermal discharge. This zone will be near MBI's Location MPMZ (MBI 2017).
3	1.0-2.0	Far-field zone within the lower reaches of Robinson Creek. This zone will be near MBI's Location RC09 (MBI 2017).

An attempt will be made to establish zones like those sampled for the 316(a) demonstration; however, they will ultimately be configured based on the thermal discharge and available habitat at the time of sample collection. Sampling zones will be documented via a hand-held Global Position System (GPS). Fish surveys will be conducted mid-summer (August) and fall (late September or early October) 2022 with the mid-summer event conducted during the bioassay study, at or near the midpoint of the study. Sampling during summer and fall will capture those

seasons when water temperatures are warmest and stream flow is lowest, compared to other seasons, and therefore represent worst case conditions. EA has been issued Scientific Collection Permits and an Endangered and Threatened Species Permit by the IDNR that includes Robinson Creek (Appendix D).



**Figure 4. Aerial View of the Robinson Creek Field Study Sampling Zones**

### 2.2.1 Robinson Creek Fish Health Specimens

To minimize stress, seining will be conducted to collect the specimens that will be evaluated for stress and health markers. Depending on habitat, either a 30-ft bag seine with 1/8-inch Ace mesh or a 10-ft straight seine with 1/8-inch Ace mesh will be used. Sampling will be conducted for up to 90 minutes at each zone, depending on the number of target specimens collected. Based on 316(a) demonstration fish community data from Robinson Creek, target fish species will likely be Creek Chub (*Semotilus atromaculatus*), Silverjaw Minnow (*Ericymba buccata*), Spottfin Shiner (*Cyprinella spiloptera*), or Bluntnose Minnow (*Pimephales notatus*). Only adult male specimens will be retained for analyses. Details of the field and laboratory analysis for fish stress and health markers as well as lesion bacterial sample collection are provided in Section 2.3.

### 2.2.2 Robinson Creek DELT Anomaly Assessment

In order to characterize and compare the incidence of DELTs among the three sampling locations, a standardized 200-meter long zone will be electrofished at each location after the health and condition specimens have been collected. If specific conductance is below 2000  $\mu\text{S}/\text{cm}$  at a given location, electrofishing will be conducted using a longline or pram method. A Smith-Root 1.5 KVA control box will provide pulsed DC output powered by a 2,000-watt generator. If specific conductance is  $\geq 2000$   $\mu\text{S}/\text{cm}$ , a Smith-Root VVP-15 electrofisher will be utilized, powered by a 5,000-watt generator. In either case, one crew member will primarily operate an electrified probe while another will collect the stunned fish and monitor the electrofishing system. A barrier net (seine) will be deployed across the entire width of Robinson Creek immediately upstream of the MPC thermal discharge Outfall 001 prior to sampling to prevent fish movement between the upstream ambient zone and the downstream near-field zone.

All fish collected will be identified to species, counted, and examined for DELT anomalies. This information will be recorded on a project-specific fish sampling data sheet (Appendix E). The incidence of DELT anomalies will be recorded following procedures outlined by Ohio EPA (2015). Fish identifications will be made using *An Atlas of Illinois fishes: 150 Years of Change* (Metze et al. 2022), and scientific nomenclature will follow Metze et al. (2022) and Van der Laan et al. (2022).

No specimens collected by electrofishing will be analyzed for the health and condition bloodwork indices because electrofishing is very stressful to fish. However, all fish that display lesions will have the lesion swabbed to identify and quantify the local bacteria present.

In-situ water quality measurements of water temperature, DO, specific conductance, and pH will be collected at mid-depth at each sampling zone. Water clarity will be measured at each zone using a Secchi disk, depending on depth. These physicochemical measurements will also be recorded on the project-specific fish sampling data sheet (Appendix E). The suggested IDNR study indicates that water chemistry samples be collected during field sampling. Therefore, water chemistry samples will be collected for analysis at Day 0, Day 30, and Day 60 for the Control, Upstream, Downstream, and Effluent sources.

## **2.3 FISH HEALTH ASSESSMENT**

Randomly selected bioassay test fish and select species of field collected wild fish will be sampled to analyze blood stress and health markers that will consist of plasma cortisol, white blood cell counts, oxidative stress, and indices of nutrition. In addition, throughout the bioassay and during each field sampling trip, fish that display lesions will have the lesion swabbed to identify and quantify the local bacteria present.

### **2.3.1 Bleeding, Necropsy, and Sample Identification**

#### Recording of Holding Conditions:

- We will monitor water temperature and DO in the coolers used to transport the fish.
- A data logger which will record temperature every 5 min will be placed in every cooler used to transport the fish. Coolers will also be kept aerated using battery-operated air pumps.

#### Bleeding:

- Make sure you have all supplies needed for bleeding:
  - Pre-weighed MS222 and sodium bicarbonate powders for anesthesia and buckets to anesthetize fish
  - Measuring board
  - Digital scale (0.001 g and 2000 g)
  - Blades
  - Capillary tubes and clay
  - Pre-labeled 15 mL conical tubes for storing capillary tubes

- Syringes and needles
- Green tops
- Trash bags
- Cooler with ice
- Sharpies and pencils
- Sharps box
- Put disposable gloves on.
- In order to anesthetize the fish, empty contents from one of the vials containing powered tricaine methane sulfonate (MS-222) buffered with equal amounts of sodium bicarbonate and targeting a 150 mg/L concentration in the anesthesia container. Mix well.
- For FHM, anesthetize fish, one at a time, in a 1 L plastic container filled halfway. For larger fish, use a 5-gal bucket filled  $\frac{3}{4}$  of the way.
- Wait for ~ 5 minutes until the fish can't maintain equilibrium and opercular movement slows down. Net the fish out and pat excess water with paper towels.
- Measure (Total Length in mm) with ruler.
- Weigh whole fish in digital scale (in mg).
- Place the fish on its side on the dissecting board. With one hand hold the fish head down and with the other make a single, quick incision in the peduncle area with a blade.
- Have three capillary tubes ready to use to collect blood by capillary action. Fill tubes no more than ~ 90% of their capacity. Seal one end with clay. Place tubes inside a pre-labeled 15-mL conical tube. Place conical tube in cooler and keep on ice until centrifuging for collection of plasma.
- If larger than 50 grams (g), fish will be bled from the caudal peduncle using 1 mL syringes fitted with 21 or 22 gauge 1 to 1  $\frac{1}{2}$ -inch needles. Place the fish on its side on the dissecting board and have a second person help hold the fish down.
- Insert needle bevel up. If bleeding is unsuccessful, you can turn your fish around and try bleeding again from the other side or from the ventral area. Apply gentle suction with the syringe and collect approximately 1 mL of blood.
- Make sure you use a new needle and a new syringe if you get any blood in it or it will clot.
- Without the needle attached to the syringe, dispense blood into the heparinized plastic tube (green top). Label your tube with Fish ID # and treatment. These are not permanent labels, so you do not need to write all the info.
- Mix gently for at least one minute!! Place in cooler with ice.
- Dispose of all blades, syringes and needles in the sharps box.
- Place capillary tubes in holding containers (in groups of 4) and spin in the field at 13,800  $\times$  g for 5 minutes.
- Break the capillary tube at the plasma line and using a 1 mL syringe, blow out the plasma into a pre-labeled cryovial. Pool all the plasma from one fish into each tube.
- Immediately place in a cooler with dry ice and store at -80°C at Purdue until processed for cortisol.

Necropsy:

- Make sure you have all supplies needed for necropsies:
  - Pre-weighed MS222 and sodium bicarbonate powders for euthanasia and buckets to euthanize fish
  - Dissecting tray and tools
  - Digital scale for weighing organs (0.001 g)
  - Pre-labeled tubes with RNA later for storing livers for gene expression
  - Empty pre-labeled tubes for storing livers for lipid content
  - Squirt bottle with 75% ETOH
  - Culture swabs
  - Paper towels
  - Trash bags
  - Paper envelopes for fish scale
  - Cooler with ice
  - Sharps box
- Put disposable gloves on.
- In order to euthanize the fish, empty contents from one of the vials containing powered tricaine methane sulfonate (MS-222) buffered with equal amounts of sodium bicarbonate and targeting a 500 mg/L concentration in the euthanasia bucket. Mix well.
- Place fish in euthanasia solution until no opercular movements are observed.
- Net the fish from the euthanasia bucket and place on a dissecting tray.
- Evaluate the external surface and note the general body condition of the fish, identify and note lesions on the skin, fins, and eyes.
- Using a bacterial culture swab, gently swab over erosions, ulcers, and areas that look abnormal (e.g., abnormal color, missing scales). Take note of where samples were collected.
- Label swab with Fish ID # and treatment. These are not permanent labels, so you do not need to write all the info.
- Place swab in cooler with ice.
- Proceed to open the abdominal cavity by cutting along the ventral midline from the gills to the anus. Remove the liver and split into two vials, one for lipid (which has no fixative) and one for gene expression which contains RNA later.
- Place sample in the cooler with ice.
- Place sample with no fixative in cooler with dry ice.
- Collect testes and weigh them.
- For wild fish, determine sex by macroscopically examining the gonads. If large enough, dissect and weigh.
- For wild fish, collect a dozen scales above and below the lateral line and save in paper envelopes.
- For FHM and small fish, place the remaining carcass in a pre-labeled Ziplock bag in cooler with dry ice. Store samples at  $-80^{\circ}\text{C}$  upon arrival at Purdue.

- For larger fish, collect fin samples (caudal, dorsal and pectoral) and place in pre-labeled bags in cooler with dry ice. Store samples at  $-80^{\circ}\text{C}$  upon arrival at Purdue.
- Discard fish remains in a garbage bag. Bring back to Purdue for proper disposal.
- Change gloves and clean dissecting board and tools with 75% ETOH in between fish.

Each sample will be assigned a unique alphanumeric identifier upon collection, SPP-#-LOCAL-ddMMM-FHI, where: SPP denotes the fish species (Table 4); # represents fish ID; LOCAL identifies where the species is collected (Table 4); ddMMM (e.g., 08AUG) denotes when the sample is collected; and FHI is the fish health index to be analyzed (Table 4).

**Table 4. Fish Health Index Sample Codes**

Species <sup>(a)</sup>	Locale	Fish Health Index
Fathead Minnow (FHM)	20°C Control (20CON)	Plasma Cortisol (COR)
Bluntnose Minnow (BNM)	20°C Upstream (20UPS)	White Blood Cell count (WBC)
Creek Chub (CCH)	20°C Effluent (20EFF)	Oxidative Stress (OXS)
Silverjaw Minnow (SJM)	20°C Downstream (20DNS)	Nutritional Condition (LIP)
Spotfin Shiner (SFS)	30°C Control (30CON)	Bacteria Culture (BAC)
	30°C Upstream (30UPS)	
	30°C Effluent (30EFF)	
	30°C Downstream (30DNS)	
	Zone 1 Robinson Creek (Z1ROB)	
	Zone 2 Robinson Creek (Z2ROB)	
	Zone 3 Robinson Creek (Z3ROB)	

(a) Additional species will be added, as necessary.

For example, if blood is drawn from the third Fathead Minnow processed on 15 August 2022 from the 30°C effluent exposure tanks, the sample identification code would be: FHM-3-30EFF-15AUG-COR.

## 2.3.2 Fish Health Indices

### 2.3.2.1 Plasma Cortisol

Once samples are received at the University of Purdue Laboratory, capillary tubes will be spun in a microhematocrit centrifuge at  $13,800 \times g$  for 15 minutes and plasma collected and stored in pre-labeled cryovials which will be flashed frozen in liquid nitrogen and stored at  $-80^{\circ}\text{C}$  at the Purdue laboratory until processed for cortisol analyses.

Cortisol levels will be quantified using a cortisol Enzyme-Linked Immunosorbent Assay (ELISA) kit according to the manufacturer's instructions. A kit sold by Salimetrics will be used, as it has worked for Zebrafish plasma with a sensitivity of less than  $0.007 \mu\text{g}/\text{dL}$  (Grzelak et al. 2017). The kit requires a total of  $25 \mu\text{L}$  plasma. However, samples will be run in duplicate using a plate reader located in the Aquatic Molecular Laboratory at Purdue University. Therefore, a total of  $50 \mu\text{L}$  of plasma will be necessary to run one sample in duplicate, which should not be an issue for fish greater than 50 g. For the bioassay Fathead Minnows in particular, plasma will be

pooled from three individuals to obtain one capillary tube/sample (50  $\mu$ L whole blood total). Assuming that approximately 50% of each blood sample is made up of red blood cells, these three fish should provide enough plasma to run this assay and an additional approximately 20  $\mu$ L of blood to quantify white blood cells.

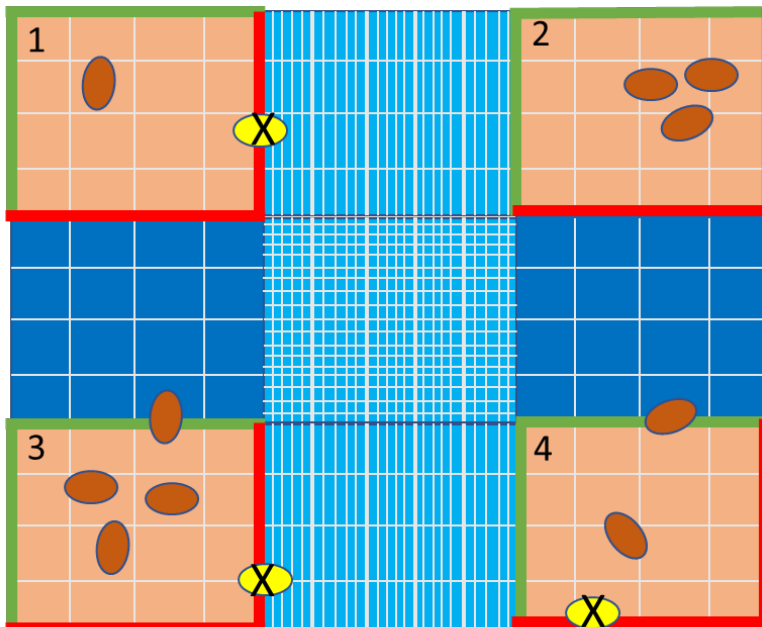
### 2.3.2.2 White Blood Cell Counts

#### Unopette Method:

The Unopette® uses a disposable diluting pipette system that provides a convenient, precise, and accurate method for obtaining a white blood cell (WBC) count. The diluent lyses the red blood cells but preserves the WBCs.

- **Clean the hemocytometer with 75% ETOH and dry with Kim wipes. Place coverslip on top.**
- **Fill up the small capillary tube that comes with the kit with whole blood and insert into vial with dye.**
- **Mix for a few seconds.**
- **Collect a sample using the larger capillary tube that comes with the kit and load it into the hemocytometer.**
- **Using a microscope, focus on the grid lines of the counting area with a 4-10x objective.**
- Out of the 9 squares, the 4 corner ones are used for WBC count (Figure 5).
- **Count the cells in one set of 16 squares (1 $\times$ 1 mm square area; the orange area). You should set a counting rule and begin counting at the top left (#1) and proceed through the 16 small squares to #2, #3 and #4.**
- Multiply the total number of cells counted by 50 and report in number of **WBCs/mm<sup>3</sup>**.





**Figure 5.** View of cell counting chambers in hemocytometer. For white blood cells, count only the four corners (orange). Only count cells that fall on the green line, not on the red line. Repeat counts if the difference between two replicates is  $> 15\%$ .

### **Blood Smears:**

Blood smears will also be made as a secondary method to quantify WBCs.

- a. Make two slides per fish. Using a pencil (not a pen which will rub off when fixing slide with methanol) label the slide on the frosted area. Use the labelling system already described.
- b. Using a capillary tube or a fine tip disposable pipette, place a small drop of blood at the edge of a clean microscope slide (slide A)
- c. Hold another clean slide (slide B or spreader slide) with thumb and index finger at  $45^\circ$  angle to slide A. Keeping the same angle, slowly move slide B toward the blood and contact these two slides with the blood. Spread the blood across to the edges of these two slides. Make sure that the  $45^\circ$  angle wedge shape constructed with two slides is filled with the blood before you spread slide B over slide A.
- d. Still holding slide B in a  $45^\circ$  angle, move the slide toward the end of slide A to make a thin even blood smear on slide A. This should be done in one movement without stop.
- e. Air dry slides and proceed to fix and stain using the kit provided. The kit consists of one fixative (methanol) and two different stains. Insert each slide into each solution 3 times. Tap slide on paper towel in between stains. Carefully wash off excess stain with water in the sink.

- f. Let slide dry and examine under the light microscope from 10 to 100X. Use immersion oil for a better examination of cells.
- g. For a WBC differential count, a total of 100 WBCs need to be counted and categorized into either lymphocytes or granulocytes (i.e., neutrophils, eosinophils & basophils).

### 2.3.2.3 Oxidative Stress

The expression of three key genes in oxidative stress will be measured: Superoxide dismutase (*sod*), catalase (*cat*) and glutathione-s-transferase (*gst*) as these have been reported as sensitive genes in response to oxidative stress in fish (Salnino et al. 2009). Expression of cytochrome P4501A (*cyp1a*) will also be quantified as an excellent biomarker of exposure to a wide range of pollutants. Gene expression will be quantified using standard qPCR protocols developed at Purdue University (e.g., Godfrey et al. 2017). Either GAPDH and/or beta-actin will be used as reference genes. If the expression of reference genes is too variable, additional genes will be selected and their combined expression may be used as the threshold to which results are compared. Primers for Fathead Minnow have been published for all of these genes (Bertucci et al. 2020).

### 2.3.2.4 Nutritional Condition

The quantity of total lipids in liver samples will be used to assess nutritional condition. A standard gravimetric method will be used as adopted for small liver (biopsy) samples of less than 100 mg, as described in Starke et al. (2010). An aliquot (2 mL) of a 3:2 mixture (vol/vol) of hexane and isopropanol will be added to each sample and after 24 hours at 20°C, the supernatant will be removed, weighed, and total lipid content determined gravimetrically as mg/g of liver.

### 2.3.2.5 Lesion Swabbing for Bacteria Culture

Using sterile swabs and avoiding cross contamination, lesions will be swabbed using sterile pipettes, which contain a culture media that preserves the microorganisms that could be present. Swabs will be kept on ice and the same day, plated on 2% blood agar plates and incubated at 20°C to 25°C for 2 to 3 days. Colonies will be counted and a subset saved for DNA analysis using a “shot-gun” genomics approach. A maximum of 60 samples will be cultured for the bioassay and field studies, respectively.

In order to characterize microbial taxonomic composition, a subset of microbial DNA directly extracted from plates will be submitted for sequencing using the 16S rRNA gene amplification method, targeting the V3/V4 region of the gene. Amplification products will be sequenced on an Illumina MiSeq platform (300 bp paired-end raw reads) either at Purdue’s Genomics Core or at a commercial lab.

- a. Lesions will be cultured on two different media (MacConkey and Levine Eosin Methylene Blue) to provide ideal culture conditions for a wide range of bacteria.
- b. Working under the biosafety cabinet in FORS 119, inoculate culture plates with swab samples.

- c. Hold the swab in one hand and lift the lid of the culture petri dish with the other. Use the lid of the petri dish to protect the agar from aerial contamination.
- d. Drag the swab in a zig-zag pattern until all surface of the plate is covered,
- e. Place lid back on petri dish and using sharpie, label the bottom of plate with your initials, date and time.
- f. Incubate plates in an inverted position at 25°C for 120 hours.
- g. Check for colony development every 24 h. Count colonies.
- h. Collect samples for DNA sequencing from a representative number of colonies and store in pre-labeled cryovials in the -80°C freezer.

### 2.3.2.6 Fish Health Assessment Sample Summary

Bioassay test specimens will be collected for analysis at the beginning, midpoint, and end of the study. Since the specimens collected at the beginning of the 60-Day study were collected prior to exposure to any treatments, five specimens from each treatment were collected. For the midpoint and end of study collections, cortisol will be sampled in five replicates for each water type and temperature exposure (40 samples per sampling period, 120 samples for the midpoint and end periods). Additionally, three types of blood and tissue samples will be collected in five replicates for each water type and temperature exposure (40 samples per sampling period, 120 samples for the midpoint and end periods). These samples will consist of white blood cell counts (leukocytes) to quantify infection levels and immune function, tissue samples to quantify oxidative stress, and liver samples to assess nutritional condition using total lipids.

As described above, field collections for health and stress markers will be conducted by seining during summer and fall. For each location and sampling event, fish health and condition will be assessed by collecting 10 replicate plasma cortisol samples (up to three fish per sample) and an additional 10 replicate fish samples (one fish per sample) for white blood cell counts, oxidative stress, and nutritional condition. In addition, fish health specimens collected by seining and all fish collected electrofishing will be examined for DELTs. Fish that display lesions will have the lesions swabbed to identify and quantify the local bacteria present. Although the number of fish that exhibit lesions is unknown, we estimate that bacterial swabs could be collected from up to 60 bioassay test fish and 60 wild caught fish. Table 5 summarizes the number and types of samples that will be laboratory analyzed.

**Table 5. Summary of the Number of Samples and Fish to be Analyzed for the Fish Health Assessment Indices**

Study Type	Response	Number of Samples Needed/Estimated*	Estimated Number of Fish Needed	Design
Bioassay	Plasma cortisol	120	360	2 temperatures x 3 sampling periods x 4 water types x 5 replicate samples
	White blood cell counts	120	120	
	Liver oxidative stress	120		
	Liver total lipids	120		
	Bacterial swabs*	60*	60	All fish with lesions

Field	Plasma cortisol	60	90	3 field zones x 2 sampling events x 1 species x 10 replicate samples
	White blood cell numbers	60	60	
	Liver oxidative stress	60		
	Liver total lipids	60		
	Bacteria swabs*	60*	60	All fish with lesions
<b>Total</b>		<b>840</b>	<b>750</b>	

### **3. DATA ANALYSIS AND REPORTING**

#### **3.1 ONSITE THERMAL BIOASSAY**

Tabular and/or graphical summaries will be provided in the report for:

- Water chemistry results from each of the three collection periods, including comparisons with results from the 316(a) study and Illinois General Use Water Quality Standards at 35 Ill. Adm. Code 302, Subpart B;
- Water temperature, DO, pH, and specific conductance of each test vessel in terms of daily minimum, mean, and maximum values;
- Daily test organism observations from each test vessel in terms of the presence or absence of DELTs and the number of dead organisms; and
- Fish swabbed and submitted for bacterial analysis.

Narrative descriptions of these results will be presented in the report along with a detailed methods section that documents all quality assurance and quality control procedures utilized (see Section 4). Any deviations or nonconformances will be documented and discussed with respect to their impact on the thermal bioassay results.

#### **3.2 ROBINSON CREEK DELT ANOMALIES ASSESSMENT**

Field data will be entered into a Microsoft Excel spreadsheet and then exported into a SAS (Version 9.2) database. Electrofishing data will be reported as number, catch-per-unit-effort (CPE, number per 300 m), and percent abundance for each species segregated by sampling zone and sampling period. DELT anomaly data will be presented as the number and percent afflicted by species and for species combined and compared spatially and temporally. The DELT anomaly data will also be compared to the 2016 results as appropriate. These results will be discussed in the final report, which will also include a detailed description of the sampling methodologies along with quality assurance and control procedures (see Section 4). The raw data will be included in an appendix.

#### **3.3 FISH HEALTH ASSESSMENT**

Tabular and/or graphical and statistical summaries will be provided in the report for (sample sizes provided under Table 5):

- Fish body sizes (weight and total length);
- Plasma cortisol concentrations;
- Total number of white blood cells;
- Expression of hepatic genes related to oxidative stress; and
- Total lipids in liver.

All raw data will be provided in the form of Excel files and analyzed using R (2022.07.01). Summary statistics will include mean, standard error of the mean, and ranges. For the bioassays, means will be compared between treatments and controls for each time point. Means of feral fish will be compared across sites for each of the time points. In the final report, a table with all the statistical results, including  $p$  and  $F$  values, will be provided for each statistical analysis performed.

## 4. QUALITY CONTROL

This study plan provides EA and Purdue University staff (the project team) with guidance regarding sampling methodologies, the equipment and supplies required, specifications regarding acceptable calibration intervals and procedures for various field and laboratory equipment, communication, quality control and assurance measures, and health and safety requirements. It also establishes various other protocols to be followed throughout this project.

In accordance with EA's Corporate Quality Management Plan (the master document for all disciplines at EA), study plans efficiently and effectively promote quality through consistency. This study plan is a project-specific document that integrates the methodologies and guidance with detailed specifications. It ensures that the study objectives will be met and that the integrity of the project team will be maintained. It also allows each staff member to understand his or her duties and responsibilities.

### 4.1 EQUIPMENT CALIBRATION

#### 4.1.1 Measuring Boards

Measuring boards and rulers used to determine lengths of fish are calibrated once after purchase, manufacture, or repair. The measuring board is calibrated with a ruler or tape that is certified by the manufacturer to be traceable to the National Bureau of Standards (NBS). Ten randomly selected points between 20 and 600 mm are visually checked against the standard ruler. Only those measuring boards that are within the stated accuracy of the standard ruler ( $\pm 1.5$  mm) are used. Measuring boards that are outside the accuracy of the standard ruler are discarded. The results of the calibration are entered on a Calibration Record Form that resides in an equipment-specific file folder.

#### 4.1.2 Measuring Scales

Spring scales are calibrated semi-annually by weighing 3-5 weights that are within the appropriate weight range. All weights are class T or better and are certified by the manufacturer to be traceable to the NBS. The readings are compared to the known weight and the results are recorded on a Calibration Record Form that resides in an equipment-specific file folder. Scales having less than 10 percent error are retained in service, whereas those that exceed 10 percent error are adjusted and recalibrated or removed from service and destroyed.

#### 4.1.3 Bioassay Temperature Monitoring System

The temperature monitoring system will be purchased from the Ideal Sciences and therefore tested and calibrated prior to the start of the study. It will be cross-checked daily against thermometer of the multi-probe water quality monitoring instrument used to measure DO, pH, and conductivity. These cross-checks will be documented on an Equipment Calibration Log (Attachment A in Appendix F, or equivalent).

#### 4.1.4 Water Quality Meters

Multi-probe water quality monitoring instruments will be used to measure DO, pH, and specific conductance. They will also be used to measure water temperature in Robinson Creek. The DO, pH, and specific conductance probes will be calibrated prior to coming onsite and once daily while onsite (Appendix F). The thermistors do not require calibration but will be cross-checked against a calibrated or reference thermistor at the same frequency as the other probes. The calibration standards will consist of:

- specific conductance: 1,000  $\mu\text{S}/\text{cm}$  conductivity standard;
- pH: buffer solutions of pH 7 and pH 10; and
- DO: in water-saturated air (or checked against a Winkler Titration).

The calibrations and cross-checks will be recorded on an Equipment Calibration Log or a Record of Calibration/Checking Form (Attachments A and B in Appendix F, respectively, or equivalent). The *YSI Professional Plus Calibrations Tips* document has been incorporated as Attachment C of Appendix F.

#### 4.1.5 Centrifuges

Centrifuges will be calibrated using manufacturers' instructions. Centrifuges will always be balanced to minimize vibrations.

### 4.2 FIELD SAMPLING

All project team members will be expected to have read and have on hand at all times a copy of the study plan. Experienced (30 years or more) project team scientists will conduct the Robinson Creek field surveys and be onsite to collect all fish health assessment samples, which will ensure strict adherence to the study plan, proper identification of fish captured, and sound judgment regarding the sample collection, preservation, processing, packing, shipping, and transportation procedures.

When collecting and handling fish, in order to preserve the integrity of the samples, we will maintain stress to a minimum, whether fish are collected via seining or electrofishing. When electrofishing, we will measure the water conductivity in the stream before electroshocking and adjust the electrofisher settings as needed to increase performance while at the same time, decrease potential damage to the fish. Because some of the responses we are measuring (i.e., cortisol and gene expression) can change quickly, we will process (anesthetize) fish as fast as possible after collection. The same care will be taken for laboratory fish so that fish are anesthetized as soon as collected from tanks. We will ensure all samples are properly labeled using a unique alphanumeric code that we will create for this project.



### **4.3 LABORATORY ANALYSES**

#### **4.3.1 Water Chemistry**

A laboratory will be contracted by MPC to measure the analytes listed in Table 1 for samples collected at the beginning, mid-point, and end of the DELTs bioassay. Analysis procedures will be conducted according to the contracted laboratories' Standard Operating Procedures, which will be documented in the laboratory reports.

#### **4.3.2 Fish Health**

For all tests described below, we will ensure all glassware used is properly cleaned and that pipettes and scales are properly calibrated as already described. Research grade chemicals will be used and purchased only from suppliers who guarantee purity. Any chemicals used for this work will be dated upon arrival and properly disposed of by expiration time. In general, samples will be run in duplicate and if differences between technical replicates is > 15%, samples will be re-run and a third replicate added.

#### **4.3.3 ELISA Tests for Quantifying Plasma Cortisol**

Our microtiter plate (Bio Tek Synergy HTX) reader is periodically controlled and maintained according to the specific recommendation of the suppliers. We will follow the QA/QC instructions provided by the vendor of the kits we plan to use (Salimetrics). Each plate will be run with a cortisol standard curve consisting of 6 concentrations plus two positive cortisol samples. Standard curves with  $R^2$  values of > 0.95 will be considered acceptable.

#### **4.3.4 Quantitative Polymerase Chain Reaction (qPCR)**

Our qPCR machine (CFX Connect Real-Time PCR) is periodically controlled and maintained according to specific recommendation of the suppliers. We will follow standardized protocols for RNA extraction, cDNA amplification, amplicon detection and confirmation, and use of positive and negative controls (US EPA 2004). We will ensure expression of reference genes is not significantly impacted by treatment/site of collection.

#### **4.3.5 Quantification of White Blood Cells**

Our microscope (Nikon Eclipse Ni scope with DS-Ri2 camera) is periodically controlled and maintained according to specific recommendation of the suppliers. If imaging is required, we will make sure scales are properly calibrated.

#### **4.3.6 Lipid Quantification in Livers**

We will run positive and negative controls during each batch of samples.

#### **4.4 FIELD AND LABORATORY DATA**

The project team will compare all (i.e., 100%) manually-entered field data against the hard copy field or laboratory data sheets. These comparisons will be kept as part of the project file, documented on a data processing log sheet (Table 6), and be made available to the client at their request. In addition, the comparisons will be done by an experienced scientist; data will not be checked by a non-scientist.

EA will perform a data assessment screening of the water chemistry analytical data packages and electronic data deliverables (EDDs) provided by MPC's contract laboratory. Data will be reviewed for completeness by comparing them to the chain of custody forms. Review of data usability will be accomplished by comparing the contents of the analytical data packages and QA/QC results to the requirements contained in MPC's Quality Assurance Plan and the respective analytical methods. EA will notify MPC of any deficiencies and work with the laboratory to resolve them.

We will ensure we maintain chain of custody forms for all the collections of biological samples. We will also ensure biological samples are maintained under the correct environmental conditions while in transit to the laboratories at Purdue University. A dataset will be considered final after cross-checking with field and lab-controlled forms and notebooks if needed.

Table 6. Data Processing Log Sheet

EA ENGINEERING, SCIENCE, AND TECHNOLOGY, INC., PBC

DATA PROCESSING LOG SHEET

PROJECT NAME : \_\_\_\_\_ PROJECT NO. : \_\_\_\_\_

DATA DESCRIPTION : \_\_\_\_\_ SAMPLE PERIOD : \_\_\_\_\_

<i>DATE</i>	<i>INITIALS</i>	<i>FILE NAME</i>	<i>ACTIVITY ( digacode, enter, proof, double check, etc.)</i>
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
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_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

## 5. HEALTH AND SAFETY

Since safety is of the utmost importance, no personnel will be required or instructed to work in surroundings or under conditions that are unsafe or dangerous to his or her health. Each individual team member will be responsible for complying with applicable safety requirements, wearing prescribed safety equipment, and preventing avoidable accidents.

### 5.1 ROBINSON REFINERY SITE-SPECIFIC REQUIREMENTS

The following information was obtained from the [Robinson Refinery Contractor](#) website and will be updated after onsite orientation. MPC Safety Procedure #12 *GENERAL SAFETY RULES* is provided in Appendix G.

#### 5.1.1 Prerequisites

Project team members must be enrolled in and tested by an MPC-approved Drug and Alcohol Test program, which is administered by DISA Global Solutions. They must also pass a DISA background security check and obtain a Transportation Worker Identification Credential (TWIC) card from the Transportation Security Administration.

#### 5.1.2 Site-specific Training

Project staff must take and successfully complete the Refinery-approved site-specific training prior to obtaining a Contractor Badge. Onsite training occurs in the Robinson Refinery Security Operations Center located directly east of the Refinery's Main Office Building, use the Route 33 entrance at the Wal-Mart stop light to access the Security Operations Center. Orientation is offered Monday through Friday at 7:30 am, 9:00 am, 10:15 am, 11:30 am, 1:00 pm, and 2:30 pm (central time zone). Contact Lisa Stewart to register for orientation, 618-546-5111 or [LStewart@Marathonpetroleum.com](mailto:LStewart@Marathonpetroleum.com).

Staff will obtain their refinery access ID card at the Refinery Security & Badging office located at: 400 S. Marathon Ave., Robinson, IL 62454. The following is required and will be verified by Refinery Security prior to issuing the Refinery access ID card:

- drug and alcohol testing compliance;
- background check;
- successful completion of the site-specific training; and
- a valid picture ID to receive your Refinery access ID card.

#### 5.1.3 Policies

##### 5.1.3.1 Smoking

Smoking (both regular and electronic) is permitted inside designated areas only. Smoking (both regular and electronic) is prohibited in vehicles within the refinery fence and in all MPC vehicles

at all times (Appendix G).

#### **5.1.3.2 Drug and Alcohol**

The possession of alcohol in unsealed or open containers as well as possession of unauthorized drugs on refinery property are prohibited. Closed/sealed containers of alcohol anywhere *other than* company parking lots outside the refinery fence line are also prohibited. No one under the influence of alcohol or illegal drugs is permitted in the refinery. Staff are responsible to notify the Medical Department in writing when they are taking prescription or nonprescription medicine or substance, which may impair their judgment or performance.

#### **5.1.3.3 Weapons**

Weapons and unauthorized firearms are prohibited on refinery property.

#### **5.1.3.4 Facial Hair**

Beards are prohibited within the refinery. However, EA has obtained a temporary waiver of the Marathon Petroleum Company LP Facial Hair Policy (Figure 5).

#### **5.1.3.5 Material Lifting**

When lifting objects >55 lbs. you should utilize one of the following options: 1) use two or more people to lift the load, or 2) use mechanical means of lifting (forklift, pallet jack, hand truck, etc.

#### **5.1.3.6 Spotter Usage Requirements for Vehicles**

Prior to entering process units, ensure provisions (spotters, barricades, etc.) are in place to prevent contact of the vehicle with process equipment. Consideration shall be given if a spotter will be required on roads not normally open to traffic, construction sites, or in heavily congested areas.

#### **5.1.3.7 Electronic Devices**

There are three types of Electronic Devices covered under #12 General Safety Rules:

- i. Type I – MPC Owned or Approved Devices with an MPC Approved Rugged Case.
- ii. Type II – Approved Contractor Devices with a case that meets all minimum requirements listed below & has an MPC Refining Approval Sticker obtained from the Safety Supervisor.
- iii. Type III – Personal Devices/Cell Phones.

The bioassay trailers are located within a restricted area. Therefore, project staff will use personal devices/cell phones only when inside the trailers or inside a vehicle. Cell Phone use in vehicles is limited to passengers, or when drivers are pulled over and parked at a complete stop or using a hands-free device. Cell Phones may not be used or on your person while operating a crane, man-lift, or anything similar in nature.

**Figure 5. Waiver of Facial Hair Policy**

Marathon Petroleum Company LP			
<b>Safety Procedure #21 RESPIRATORY PROTECTION</b>	Document No. 1021	Page Page 12 of 12	Revision No. 14
	Original Issue Date 02-82	Revision Date 12-6-2017	Next Revision Date 12-6-2021
	Document Custodian: Safety Control Group: Refining Operations - Refining - State Work Practices Revised: 2021-12-06 revised, incorporated in no longer revised		

Appendix C

MARATHON PETROLEUM COMPANY LP  
Refining Division

Date: 7/14/2022

WAIVER OF FACIAL HAIR POLICY

A temporary waiver of the Marathon Petroleum Company LP Facial Hair Policy is being temporarily issued to

EA Engineering (name employee and company, or company only) for

the purposes of performing the following work at the specified location within the refinery:

EA Engineering personnel will be working on site in temporary trailers set up west of the Ethanol WWTP Miller process building. EA Engineering personnel will be performing a scientific study required by the refinery H2S-H2S Permit on full, untemperatures-controlled tower that with WWTP effluent and water from Robinson Creek.

This waiver is valid for the following dates: July 2022 - October 2022

*Kathy Harrison*  
Department Manager  
Refining Division

NOTES:

- This waiver is not required for delivery personnel and visitors covered under a visitor pass, as long as they are not required to wear respiratory protection.
- This waiver must be kept with the individual to which it was issued and a copy provided to Security when entering the gate.
- The waiver can only be written up to a 12 month period.

ALL INFORMATION CONTAINED HEREIN IS UNCLASSIFIED EXCEPT WHERE SHOWN OTHERWISE. THIS COPY IS CONTROLLED BY THE COMPANY AND IS NOT TO BE DISTRIBUTED OUTSIDE THE COMPANY.

Marathon Petroleum Company LP			
<b>Safety Procedure #21 RESPIRATORY PROTECTION</b>	Document No. <b>1021</b>	Page <b>12 of 12</b>	Revision No. <b>14</b>
	Original Issue Date <b>02-02</b>	Revision Date <b>12-6-2017</b>	Next Revision Date <b>12-6-2021</b>
Document Custodian: Safety		Control Category: REF. 0808210111 - Ref - Safe Work Practices Replaces: 0801-001 Revised, superseded. For no longer required	

Appendix C

MARATHON PETROLEUM COMPANY LP  
Ethanol Refining Division

Date: 7/14/2022

WAIVER OF FACIAL HAIR POLICY

A temporary waiver of the Marathon Petroleum Company LP Facial Hair Policy is being temporarily issued to EA Engineering (name employee and company, or company only) for the purposes of performing the following work at the specified location with the urgency:

EA Engineering personnel will be working on site in temporary trailers set up west of the Plant #1 WWTMP (the process building). EA Engineering personnel will be performing a scientific study required by the refinery. H2S Permit will follow in temperature-controlled trailers that catch WWTMP effluent and water from Robinson Creek.

This waiver is valid for the following dates: JUN 2022 - October 2022

  
Kathy Wilkinson  
Department Manager  
Ethanol Refining Division

NOTES:

- This waiver is not required for delivery personnel and visitors covered under a visitor pass, as long as they are not required to wear respiratory protection.
- This waiver must be kept with the individual to which it was issued and a copy provided to Safety when entering the gate.
- The waiver can only be written up to a 12 month period.

ALLISTON: Printed copies should be issued with caution. The user of this document is responsible for ensuring that the information is current and accurate. This document is controlled under the 2002-2004 OSHA 1910.101.



#### **5.1.4 Personal Protective Equipment**

Personal protective equipment and safety devices must be used as required and must not be altered in any manner. The use of damaged or malfunctioning personal protective equipment is prohibited.

##### **5.1.4.1 Safety Glasses with Approved Side Shields (ANSI - Z87.1)**

ANSI approved safety glasses with side shields must be worn at all times within the refinery where work is being performed. This includes maintenance shop areas, the laboratory, and at designated work sites away from the refinery.

Safety glasses with side shields are not required to be worn in the following locations:

- 1) West of 2 ½ Street,
- 2) Lunch/break rooms, control rooms, or plant offices, and
- 3) Inside vehicles with enclosed cabs (windows closed).

Contact lenses may be worn in conjunction with safety glasses/side shields. Workers who wear contact lenses should inform the refinery nurse of their use. The nurse will issue hard hat stickers indicating contact use.

##### **5.1.4.2 Goggles and Face Shields**

Employees are required to have ANSI Z87.1 approved chemical splash goggles on their person (i.e., on their hard hat, in a pouch on their belt, etc.) when in process areas, the tank farm, or designated off site locations where the potential for flying debris or chemical exposure exists.

At a minimum, unless engineering controls are in place, a face shield OR goggles must be worn when disconnecting hoses when potential for pressure exists.

Goggles must be worn for the following jobs or where there is risk of debris falling into the head/face area as a result of the work:

- 1) Handling powdered, granulated or dusty materials and loose insulation. Note that if there is the need to use a dust mask or half mask particulate respirator, goggles still must also be used.
- 2) Catching hydrocarbon samples.
- 3) Using pressurized air, steam, etc. to clean equipment.
- 4) Opening or transferring chemical totes via hoses.
- 5) When performing any internal cleaning of dirt/debris in vessels, tanks, exchanger shells, furnaces, etc.

A face shield (over safety glasses) must be worn for the following jobs:

- 1) When a flying chip hazard exists (i.e., grinding, chipping such as concrete/refractory, cutting, buffing, blasting, etc.),
- 2) While grinding or buffing vessels or equipment.
- 3) When using a torch/wand to light burners on heaters or boilers.
- 4) Operating an air powered nut gun/impact wrench.
- 5) When handling/working with hot products 140° F (molten sulfur, hot residue, hot condensate/boiler feedwater, etc.).
- 6) Operating a string trimmer during lawn maintenance.
- 7) When looking into fired heaters and boilers.

A face shield AND goggles must be worn for the following jobs:

- 1) Connecting/disconnecting lines or hoses in acid or caustic service.
- 2) When catching samples in acid or caustic service.
- 3) Cleaning, draining or repairing equipment which has been in acid or caustic service and not neutralized.
- 4) Loading or unloading of acids or caustics.
- 5) Initial line breaking or opening of equipment when potential for pressure exists.
- 6) Open sampling of liquids/products above 140 degrees F (non-engineering sample systems).

#### **5.1.4.3 Safety Toe Shoes (ASTM F2413)**

ASTM approved safety toe shoes with at least a ¼” defined heel must be worn at all times within the refinery property and at designated work sites away from the refinery when work is being performed.

ASTM approved shoes are not required to be worn in the following locations:

- Lunch/break rooms, control rooms, plant offices,
- Inside vehicles,
- Employees reporting to work or leaving work provided they go directly to their work area,
- Walking directly to or from personal vehicles or offices outside process unit battery limits,
- Truck drivers and vendors making deliveries or pickups of supplies, and
- Laboratory – shoes must be made of leather, rubber, or other non-absorbing material.

#### 5.1.4.4 Head Protection (ANSI Z89.1 Type 1 Class “E”)

All employees are required to wear an ANSI Z89.1 Type 1 Class “E” approved hard hat when in process areas, tank farm, designated off site locations where work is being performed, or new construction areas.

- 1) Hard hats must be changed at a minimum of every five years from the born-on date or when damaged or showing visible signs of wear (i.e., cracks, disfigurement, UV damage, etc.).
- 2) Hard hat suspensions must be changed at least annually.
- 3) Hair length longer than the shoulders must be kept under a hardhat when working around rotating equipment.

#### 5.1.4.5 Flame Resistant (FR) Protective Clothing

These procedures must be adhered to in order to provide adequate protection for workers in areas where there are recognized fire hazards and a reasonable probability that FR could mitigate burn injuries.

1. All FR clothing base garments (shirt/pant combo and/or coveralls) shall either be inherently FR material (e.g., Nomex, PBI) or FR treated cotton and cotton blends that are certified by an independent testing agency meeting NFPA 2112.
2. Seasonal accessories (e.g., UV face masks, cold weather beanies, or hard hat liners) shall also be meet NFPA 2112. (RSP Compliance Date - January 1, 2020)
3. Garments worn underneath base layers for warmth/cooling shall be made of natural fibers such as cotton, wool, or silk. This requirement does not include underwear.

**IMPORTANT:** Base layers made from synthetic materials such as polyester (e.g., Under Armor) are Prohibited.

4. FR shirts (not including outer FR garments (e.g., coats and sweatshirts with or without hoods, etc.) shall be tucked in, buttoned up, and sleeves rolled down when in FR required areas to comply with NFPA 2113.
5. Outer FR garments (e.g., coats, bibs, and sweatshirts with or without hoods, etc.) shall be made of FR fabric and adhere to NFPA 2112 requirements that are certified by an independent testing agency.
6. NFPA 2112 daily FR work wear garments shall be worn at all times under all outer FR garments.
7. Hole watch/Fire watch vests shall comply with ASTM D6413 Flame Resistant requirements. FR Rain Wear: (RSP Compliance Date - January 1, 2020)
8. All rain wear shall comply with ASTM D6413 Flame Resistant requirements, and shall be tested and comply in accordance with:
  - a. ASTM F2733 for flash fire, and
  - b. ASTM F1891 when the risk potential of an arc flash hazard exists.

**FR Disposable Coveralls:**

9. Disposable coveralls shall be made of FR fabric and are not required to meet NFPA 112 requirements.
10. Disposable coveralls shall comply with ASTM D6413.
11. Disposable coveralls shall comply with NFPA 2113 as it pertains to the care and maintenance during use.

NOTE: Any garments soiled with hydrocarbons or visibly tattered during work activities must be removed from service and replaced.

Each employee shall be responsible for the inspection and integrity of fire-resistant garments issued to them. Employees shall routinely inspect the garments for rips, tears, holes, discoloration, function of buttons, zippers, and fabric thinning due to age and repeated washings. Damaged clothing should be repaired or replaced.

FR shall be worn by all personnel in the refinery with the following exceptions:

- 1) Employees will be allowed entry into the refinery while wearing dresses, sleeveless shirts, & short pants, west of 2nd Street and including the E&I Shop, Main Warehouse, or while riding in an enclosed vehicle to Complex / PDU / Lab break rooms.
- 2) Employees reporting to work and leaving work, provided they go directly to their work area.
- 3) In Control Rooms and offices that are outside process unit battery limits.
- 4) Inside the Warehouses, E & I Shop, Machine Shop, Welding Shop, the Garage and Firehouses provided that no threat of flash fire exist.
- 5) While in the offices, main hallways and lunch/break rooms in the Laboratory.
- 6) In new construction areas that are not in an operating unit.
- 7) On refinery roadways.

**5.1.4.6 Hand Protection**

Gloves must be worn for jobs that have the potential for hand injury. Each person when in process areas, the tank farm, or designated off site locations where the potential for hand injury exists who is required to wear fire resistant clothing shall at least have general duty work gloves conforming to ANSI/ISEA 105 Level 3 at least in the palm, fingers and thumb of the glove for general operations and maintenance work.

For tasks with the potential of impact hazards, gloves with impact protection to the back of the hand and full length of the fingers are to be worn. (e.g., work with hammers, picking up blinds/valves, hand wrenching, flange bolts, impact gun tasks, tasks where hands and fingers can be pinched between the tool and a fixed object or material)

#### 5.1.4.7 Hearing Protection

Hearing protection is required to be worn inside the operating boundary (perimeter) of all process units, including during shutdown/turnaround periods. High noise areas in the plant may be designated by a yellow stripe and/or signs stating “Caution - Ear Protection Must Be Worn In This Area”. High noise areas are also encountered around operating equipment such as vacuum trucks, compressors and operating pumps in the tank farm. Hearing protection must be worn regardless of the time spent in these areas.

#### 5.1.4.8 Life Jackets

U.S. Coast Guard-approved life jackets must be worn at all times whenever there is a danger of falling into a body of water and 100% fall protection cannot be maintained. This includes barges, floats (without handrails), rowboats, motorboats, or any other equipment in or over water. **Life jackets will be worn at all times during electrofishing activities on Robinson Creek if water is greater than three feet deep and/or fast moving.**

When wearing a life jacket or work vest it should be adjusted and the top and bottom buckles fastened. Prior to and after each use, the life jacket or work vest must be inspected for defects which would alter their strength or buoyancy. Defective units must not be used.

### 5.2 ELECTROFISHING

#### 5.2.1 Introduction

In many cases, the most effective means of collecting fish for scientific purposes is electrofishing. Electrical current is placed in the water to immobilize fish, allowing them to be collected with dip-nets. It involves the use of either AC (alternating current) or DC (direct current) to immobilize fish for capture. These two types of current have very different effects on fish. The choice of current to use is dependent on the type of study being performed and the importance of returning healthy fish to the water. For the Marathon Robinson DELTs study, electrofishing will be used to conduct select elements of the field collections on Robinson Creek.

##### 5.2.1.1 AC & DC Current

AC current typically has the most violent effect on fish. Once in the electrical field a fish will immediately “take a posture transverse to the current in such a way as to receive a minimum of voltage” (Smith-Root). This action is called oscillotaxis. Fish will be immobilized quickly and the effect will last longer than that of DC current. Great care must be taken in the collection of fish in this manner. For the Robinson Creek field collections, we will be using DC electrofishing.

With DC current, fish react in three ways: first, they line up with the direction of the electrical current, then swim toward the anode (positive electrode). This reaction is called galvanotaxis. Finally, when fish near the anode they are stunned, roll belly up, and collection becomes possible. The effects of DC current do not last as long as AC current. When the power is turned off the fish recover quickly. Mortality is far more limited than with the use of AC. This,

along with the fact that fish actually swim to the anode, makes DC current the more effective means of electrofishing.

### **5.2.1.2 Control Box**

DC current can be selected with electrofishing control box. In addition to controlling the type of current, a control box allows adjustments to how the current acts. Most equipment will allow you to select for standard or pulsed output and to vary the pulse width and frequency of pulses, which allows for more efficient collections and limits the risk and stress to fish.

The control box also allows selection of voltage output. Depending on the electrofishing system used (i.e., Smith-Root), this selector should be positioned at the lowest possible setting that allows 5-10 amps to be obtained by adjusting the pulse width and rate or a minimum of 190 volts.

Pulsed output means that the electrical current going from the system into the water comes in pulses or waves. When the pulse rate is low and the width of the field is narrow, less current is required to collect fish. This results in less stress to fish. Since conductivity of water (the ease with which an electrical charge passes through it) varies, it is necessary to have the ability to adjust the pulse rate and width for optimum collection with minimum harm to the fish being collected.

### **5.2.1.3 Conductivity**

Electrofishing works by passing electrical current through a fishes body causing the effects described above. Several factors affect the amount of current passing through the fish's body and thus, the effectiveness of electrofishing. If the conductivity of the fish's body is equal to or slightly above the conductivity of the surrounding water, the electricity will choose the path of least resistance and pass through the fish. The greater the conductivity of the fish's body in relation to the surrounding water, the greater the effect of the electricity on the fish. The conductivity of fish flesh differs among species. When shocking, you may observe catfish floating up as far as 50 ft. from the anode. At the same time, scaled fish may not succumb to the current until they actually pass within a few feet of the anode. Also, due to increased surface area, larger fish, particularly large and deep-bodied fish, tend to receive a larger charge of electricity than do smaller fish.

Another factor that influences the effectiveness of electroshocking is the conductivity of the water. Pure distilled water will actually act as an insulator in an electrical current. This is because there are few electrolytes or dissolved solids to conduct the electricity. It would take a great deal of current to pass through this type of water. Conversely, the water of a typical lake or river may be very high in dissolved solids. This water will readily conduct very low amounts of current. In all cases, the conductivity of the water must be equal to or below the conductivity of the fishes body for electrofishing to be effective. It is not effective to shock in salt water because it is an electrolyte solution. The conductivity of the water is so much higher than that of a fish that an electrical current will find that the path of least resistance is actually around the fish rather than through it.

Conductivity of the water being surveyed should always be checked before attempting electrofishing. If it is very low ( $<50 \mu\text{S}/\text{cm}$ ) or extremely high ( $>4500 \mu\text{S}/\text{cm}$ ), a different type of collection should be considered. When backpack, pram (tow barge), or long line (bank unit) shocking small streams, it may actually be possible to increase the conductivity of the water by placing a block of salt upstream of the study area several hours before beginning your survey. This however, should only be considered in controlled conditions.

#### **5.2.1.4 Types of Equipment**

There are several types of electrofishing equipment available. EA typically uses boat, backpack, pram or long line units. These units differ in the type of power source used and in their application. For the Robinson Creek field collections, EA will use pram and/or long line wadeable units while wading in Robinson Creek.

Pram and long line electrofishing are designed for use in areas where boat electrofishing may not be possible or practical. Pram shocking involves the use of a power source and electroshocking unit placed in a barge or small boat. Like backpack electrofishing, the operator utilizes a hand held anode and trail behind cathode to place current in the water.

Long line electrofishing involves the use of a power source and electroshocking unit deployed on the bank. Like the other wadeable methods, the operator utilizes a hand held anode. However, the cathode is stationary, typically deployed in the middle of an electrofishing zone near the control box and power source, to place current in the water. As with the pram unit, the operator is not required to carry the power source and control box. Cables with up to 100m long allow mobility over a large section of water.

In all types of electrofishing, current is passed through the water between a positive electrode (anode) and a negative electrode (cathode). EA typically uses a boom mounted anode and the boat hull as a cathode when boat electrofishing. You may however, see different arrangements. In backpack electrofishing the anode is a hand-held probe or dipnet and the cathode is a trail behind cable. In pram shocking, the cathode may be the hull of either the barge or boat carrying the equipment, and in long line shocking the cathode is a cable or plate deployed from a bank-mounted power source.

#### **5.2.1.5 Equipment Operation**

Pram and long line shocking are slightly more hazardous than boat shocking because of the user's position in the water with the electrical charge. EA will utilize only experienced staff with several years of experience with the equipment and conducting the same type of work as will be done for the Robinson Creek field collections. For staff and visitors that are new, a field brief or field training sessions will be completed, as needed, before initiating the work.

Basically, a wadeable system is a miniaturized version of the boat electrofishing system. At least two operators are required for pram electrofishing while three operators are preferable when using the long line method. For pram shocking, the operator handles the anode, which consists of

a probe or a combination of probe and dipnet, depending on conditions. The second person monitors the equipment while assisting with the collection, transfer, and care of fish. For long line electrofishing, a third person typically maintains the cable and manages the live car.

The operator wades in an upstream direction through the water sweeping the anode 2-3 feet ahead. A thumb switch on the handle of the probe serves the same safety function as the foot switch on the boat. With a net probe, when a fish is shocked, the operator collects it with the dipnet, releases (i.e., turns off) the switch on the handle and places the fish in a bucket, live-well, or live car. If the anode is not operated with an attached net, the second person will closely follow the operator and anode with a dipnet to collect fish. When pram shocking, special attention should be paid by all crewmembers to the size of the electrical field. If the cathode is mounted on a barge, boat, or bank the electrical field will reach from that point to the anode held by the operator.

## **5.2.2 Safety**

### **5.2.2.1 Safety Awareness**

For the Robinson Creek field collections electrofishing will be performed by a trained field crews, with well-maintained equipment, electrofishing can be a very safe means to collect fish for biological study. Nonetheless, attention to safety must be paramount for all crew members in order to conduct a successful electrofishing survey. The amount of current in the water may be in excess of 250 volts. The amount of amperage generated during typical shocking operations averages 8 amps. This is enough to harm people, under certain circumstances, if the field were to come in direct skin contact with an electrical source such as a cathode, anode over a significant portion of the body (e.g., falling into and completely submerging in a strong electrical field at a close proximity to the sources mentioned or in concert with select medical conditions).

Therefore, awareness of the hazards, proper PPE, experience, and caution are paramount to the safe operation of electrofishing equipment.

### **5.2.2.2 Hazard Awareness**

Various physical hazards will potentially be present during electrofishing activities. These physical hazards may include, but may not be limited to:

- Working over, near, or in the water
- Slip, trip, and fall
- Weather
- Material handling, moving, lifting
- Fire/explosion
- Exposure (e.g., cold stress, heat stress, sun burn)
- Noise
- Electrical
- Biological (e.g., fish spine puncture wounds, poisonous insects and plants)



### 5.2.2.3 Safety Rules

Always follow the manufacturer's instructions when installing or operating electrical equipment. It is each crew leader's responsibility to familiarize crew members with the equipment and how to operate it. Furthermore, it is the responsibility of each crew member to assure that others are following proper procedures. **If EA staff are asked to do something that they feel is improper or unsafe, all have the authority to refuse and stop work.** Don't depend on someone else to look out for you. Look out for yourself.

Despite all of this, as mentioned above, electroshocking surveys can be conducted in a safe manner. All that is required is proper attention to detail and the use of the safety equipment provided.

The following are the primary common-sense rules that must be followed by all crew members at all times:

1. Life jackets are required to be worn at all times when water depth is greater than three feet and/or electrofishing is being conducted in fast moving water.
2. Prior to initiating a survey, the crew leader will conduct a safety briefing to remind or instruct support personnel on basic operation, safety, and hazard awareness. Prior to electrofishing at a given site, the crew will survey the study zone for potential hazards.
3. Wear rubber gloves when operating/touching electroshocking equipment.
4. Non-breathable, chest waders will be worn by all crew members for wadeable electrofishing
5. When conducting wadeable electrofishing, all equipment in the water (e.g, nets, live cars, live wells, buckets) must be non-conductive, insulated, and/or isolated.
6. Due to the conductive nature and added weight, steel-toed boots and/or weight belts must never be worn while electrofishing.
7. Lug-soled boots are appropriate when wading in soft and or fine substrates (e.g., silt, much, gravel, cobble). However, large and firm substrates (e.g., bedrock, boulder, large cobble) may be especially slippery and may require felt-soled wading boots or corkers to safely wade.
8. Never touch a loose wire or make an adjustment while unit is in operation. Rubber gloves must be worn, safety switches must be released, and the control box turned off before making any output adjustments. For all other system adjustments, beyond source, output, and other fine tuning at the control box, the power source and system must be shut-down, completely.
9. Always use safety switches. Never disable a safety switch or use equipment with an inoperable safety switch.
10. Never over-extend yourself when netting fish.
11. When wading, walk deliberately and carefully with a shuffling, wide stance to avoid unseen trip hazards.
12. Communicate hazards to fellow wading crew members. Each crew member has limits to their view. Don't assume everyone sees what you see. If noise level restricts normal conversation, establish hand signals.

13. Never place your bare hand in the water.
14. Look up from the water frequently to assure that overhanging branches or other items don't pose a risk.
15. If necessary, particularly for boat or pram operated equipment, wear hearing protection.
16. Maintain the equipment through routine maintenance checks. If repairs are needed, get them fixed immediately. Don't wait for the next person to do it.
17. Life jackets are not recommended for wadeable electrofishing in shallow water (i.e., less than three feet deep) as they restrict movement and may contribute to heat stress. In doing so, life jackets with shallow water, wadeable electrofishing actually present a greater hazard than wading without a life jacket.
18. Cold weather, dress warmly in layers of reasonably tight-fitting materials. Additional protections may include glove liners, hats, hand and foot warmers. For warm weather, light colored and light weight synthetic, quick dry fabrics should be worn. Maintain hydration and use sunscreen liberally to protect from sun burn.

Robinson Creek is a shallow stream with most working areas no deeper than two feet. In the event a staff member was to fall, in most cases, they can self-recover to their feet or be assisted by other electrofishing crew members. In the event a staff member becomes incapacitated, first, get the individual's face out of the water, check condition, and, if necessary, call 911 for emergency assistance. As needed, perform first aid and CPR.

### **5.3 WEATHER HAZARDS**

Weather conditions will be taken into consideration during each sampling effort. Heavy rains, electrical storms, high winds, and extreme temperatures, for example, may create extremely dangerous situations for employees. Inclement weather may also impair equipment performance. Whenever unfavorable conditions arise, the Crew Leader will evaluate both the safety hazards and ability of the employees to effectively perform given tasks under such conditions. Outdoor work will be suspended during thunderstorms, tornadoes, hurricanes, or other severe weather events. Weather conditions will be monitored via cell phone to identify the approach of severe weather situations.

### **5.4 HEAT STRESS**

Prolonged exposure to heat can result in heat rash (prickly heat), heat cramps, heat exhaustion, or heat stroke. Heat stroke is life threatening and requires immediate professional medical attention. An overview of these heat-induced illnesses and proper preventative actions are described below.

#### **5.4.1 Heat Rash (Prickly Heat)**

Heat rash, which is commonly observed in tropical climates, is a painful temporary condition caused by clogged sweat pores, typically from sleeping in hot, humid quarters. Heat rash appears as tiny red bumps on the skin and can impair sweating, resulting in diminished heat tolerance.

Heat rash can usually be cured by providing cool sleeping quarters; body powder may also help absorb moisture.

#### **5.4.2 Heat Cramps**

Heat cramps are characterized by painful intermittent spasms of the voluntary muscles following hard physical work in a hot environment. Heat cramps usually occur after heavy sweating and often begin towards the end of the workday. The cramps are caused by a loss of electrolytes, principally salt. This results in fluids leaving the blood and collecting in muscle tissue, resulting in painful spasms. Treatment consists of increased ingestion of commercially available electrolytic “sports” drinks (because of individual sensitivity, it is best to dilute by doubling the amount of water required by package directions or add water to the liquid form).

#### **5.4.3 Heat Exhaustion**

This condition is characterized by profuse sweating, weakness, low blood pressure, rapid pulse, dizziness, and frequently nausea and/or headache. The skin is cool and clammy and appears pale. The body core temperature is normal or depressed. Victim may faint and/or vomit. This is the most common work-related heat illness, and usually occurs after an extended period of work – look for signs of onset after lunch – an employee may suddenly need to sit down, feel faint, weak, or nauseated.

First aid consists of placing the victim in a cool area, loosening clothing, placing in a head-low (shock prevention) position, and providing rest and plenty of fluids. Any worker who is a victim of heat exhaustion may not be exposed to a hot working environment for an absolute minimum of 24 hours, and if fainting has occurred, the victim should not return to any work until authorized by a physician.

#### **5.4.4 Heat Stroke**

This is the most serious heat disorder, is life threatening, and is a true medical emergency. It results when the body’s heat dissipating system is overwhelmed and shuts down (thermoregulatory failure). Heat stroke results in a continual rise in the victim’s deep core body temperature, which is fatal if not checked. The symptoms are hot, dry, flushed skin, elevated body core temperature, convulsions, delirium, unconsciousness, and possibly death.

First aid consists of immediately moving victim to a cool area; cool the body rapidly by immersion in cool (not cold) water or sponging the body with cool water; treat for shock and obtain immediate medical assistance. Treatment response time is critical when assisting a victim of heat stroke! Do not give coffee, tea, or alcoholic beverages.

#### **5.4.5 Preventative Measures**

Unfortunately, there are no known PPE to prevent heat-related illnesses. However, some preventative measures to avoid heat stress include:

- Frequent resting in cool or shaded areas,
- Consumption of large quantities of potable water or diluted electrolyte beverages, following the suggested hydration target in Table 7:

**Table 7. Hydration Targets based on Air Temperature and Time Periods between Breaks**

Temperature	Work Level	Maximum Minutes Worked Between Hydration Breaks	Hydration Target
<80°F	Normal	--	8-12 oz./hr.
80-85°F	Normal	--	8-16 oz./hr.
86-90°F	Normal	50	12-20 oz./hr.
91-95°F	Normal	45	16-24 oz./hr.
>96°F	Normal	40	24-32 oz./hr.

- Following a work/rest regiment from Table 8:

**Table 8. Work/Rest Schedule based on Air Temperature**

Ambient Temperature	Work (hours)	Rest (minutes)
70°F	3	15
75°F	2½	15
80°F	2	15
85°F	1½	15
90°F	1	15

Other factors, such as a worker's acclimatization, level of physical fitness, and age, may increase or decrease his/her susceptibility to heat stress. Before assigning a task to an individual worker, these factors will be considered to ensure that the task will not endanger the worker's health.

If a heat-related illness is suspected or observed, the affected person must be moved to a cool or shaded area and given plenty of liquids to consume. If symptoms of a heat stroke are observed, the victim will be cooled immediately and treated as a medical emergency. Liquids will be readily available to ensure that workers stay hydrated.

## 5.5 BIOLOGICAL HAZARDS – INSECT BITES/STINGS

Protective outer clothing such as gloves, hard hats, and coveralls can reduce the potential for insect bites and stings. Insect bite symptoms may include redness, rash, swelling, chills, fever, diarrhea, and vomiting. Any worker who has been bitten or stung and shows symptoms of a severe reaction should seek medical assistance immediately. Workers who know of their allergies to insects should advise their supervisor prior to field activities and should carry an antidote kit, if necessary.

When working in areas near heavy vegetation (possibly the riverbank) and to prevent contact with disease-carrying ticks, workers should wear long-sleeved shirts, long pants, and boots that extend above the ankle with socks pulled over pant cuffs or with pants legs taped to boots. Insect repellent is also an effective means of tick control. Workers should check clothing, skin,

and hair for the presence of ticks periodically and thoroughly at the end of each workday. If a tick attaches to the body, it should be removed by gently tugging with tweezers where the mouth enters the skin. The tick should not be killed prior to removal.

## **5.6 BIOLOGICAL HAZARDS – FISH**

Care must be taken and the proper PPE used when handling certain fish species to avoid getting cut, spined, or bitten. Proper handling of fish will be performed by experienced project personnel to reduce the likelihood of injury. Any worker that gets injured from a fish should seek medical attention immediately.

## **5.7 POISON IVY AND RELATED PLANTS**

Poison ivy, poison oak, and poison sumac have poisonous sap (urushiol) in their roots, stems, leaves, and fruits. The urushiol may be deposited on the skin by direct contact with the plant or by contact with contaminated objects, such as clothing, shoes, tools, and animals. Preventative measures include: wear long-sleeved shirts and long pants tucked into boots; wear cloth or leather gloves; apply barrier creams (e.g., Ivy Block) to exposed skin; and be able to identify poison ivy, oak, and sumac plants. If you are exposed, according to the U.S. Food and Drug Administration (FDA), you should quickly (within 10 minutes): 1) cleanse exposed areas with rubbing alcohol; 2) wash the exposed areas with water only (no soap yet, since soap can move the urushiol, which is the oil from the poison ivy that triggers the rash, around your body and actually make the reaction worse); 3) take a shower with soap and warm water; and 4) put gloves on and wipe everything you had with you, including shoes, tools, and your clothes, with rubbing alcohol and water.

Unfortunately, if you wait more than 10 minutes, the urushiol will likely stay on your skin and trigger the poison ivy rash. You may not be able to stop it on your skin, but you might still scrub your nails and wipe off your shoes, etc., so that you do not spread the urushiol to new areas.

## **5.8 ALLERGIC REACTIONS**

When in the field, personnel may be exposed to allergens that can cause mild to severe allergic reactions. The following guidelines will explain how to help a person having an allergic reaction.

For a mild to moderate reaction:

- Calm and reassure the person having the reaction, as anxiety can worsen symptoms.
- Try to identify the allergen and have the person avoid further contact with it. If the allergic reaction is from a bee sting, scrape the stinger off the skin with something firm (such as a fingernail or plastic credit card). Do not use tweezers; squeezing the stinger will release more venom.
- If the person develops an itchy rash, apply calamine lotion and cool compresses.

Avoid medicated lotions.

- Watch the person for signs of increasing distress.
- Get medical help. For a mild reaction, a physician may recommend over-the-counter medications (such as antihistamines).

For a severe allergic reaction:

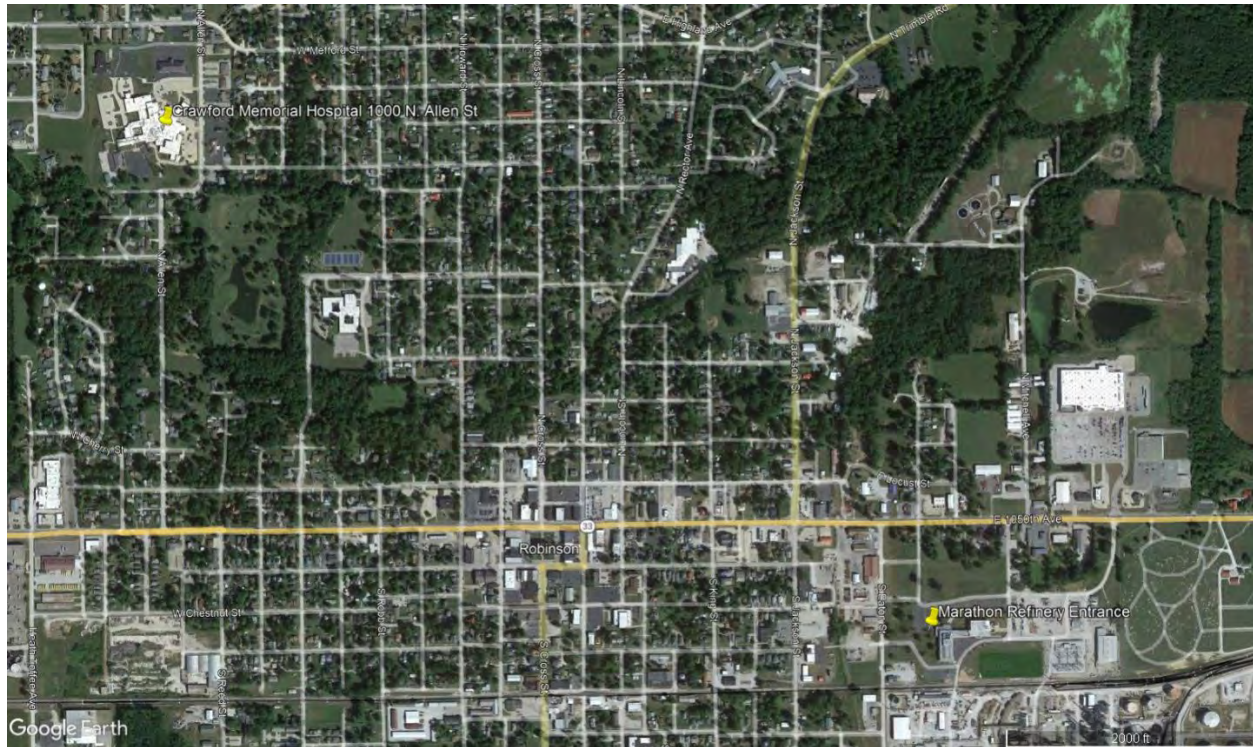
- Check the person's airway, breathing, and circulation (the ABCs of Basic Life Support). A warning sign for dangerous throat swelling is a very hoarse, whispered voice or coarse sounds when the person is breathing air in. If the victim is having difficulty breathing, is very weak, or is losing consciousness, call for emergency medical assistance.
- Calm and reassure the person.
- If the person has emergency allergy medication on hand, help the person take or inject the medication. Avoid oral medication if the person is having difficulty breathing.
- Take steps to prevent shock. Have the person lie flat, elevate the person's feet about 12- inches, and cover him or her with a coat or blanket. **DO NOT** place the person in this position if a head, neck, back, or leg injury is suspected or if it causes discomfort.

## **5.9 EMERGENCY RESPONSE PLAN**

In the event of an emergency, the information available at the time must be properly evaluated and the appropriate steps taken to implement the emergency response plan. The Crew Leader or senior onsite supervisor will assume command of the situation and call 618-544-2121 Ext. 5300 inside the refinery or 911 outside the refinery from the nearest telephone or cell phone, to notify authorities of your location (the docking point at which you will meet them), evacuate personnel as needed, and take other steps needed to gain control of the emergency.

Appropriate first aid will be given and emergency contacts will be made. Emergency situations will be handled by offsite support personnel; however, initial response and first aid will be available from qualified onsite personnel. Once the situation is under control, the Crew Leader or designee will immediately call EA's Corporate Safety and Health Officer (Rob Marcuse at 410-329-5192) and must complete an Accident/Loss Report.

The nearest hospital to the project site is Crawford Memorial Hospital, located at 1000 N. Allen Street, Robinson, Illinois 62454 (618-544-3131).



**Figure 6. Location of Crawford Memorial Hospital and MPC Entrance.**

### **5.10 CORONAVIRUS DISEASE 2019 (COVID-19)**

All EA staff will be fully vaccinated against COVID-19 prior to arrival on-site. EA staff will adhere to current state and/or local PPE and social distancing requirements, as necessary. Appropriate PPE, including face masks and hand sanitizer, will be available to EA staff as needed.

**6. COMMUNICATION AND CONTACT INFORMATION**

<b>LOCAL EMERGENCY TELEPHONE NUMBERS</b>	
Crawford Co. Sherriff	911 or (618) 546-1515
Robinson Police Department (Robinson, IL)	911 or (618) 544-2217
Robinson Township Fire Dept. (Robinson, IL)	911 or (618) 544-2955
Crawford Memorial Hospital (Robinson, IL)	(618)-544-3131
Poison Control Center	(800) 492-2414 or (800) 222-1222
<b>Region 5 Department of Natural Resources Personnel (618) 435-8138</b>	
Boone LaHood - Fisheries Biologist	Office (O): (618) 393-6732
Logan Willand - Area Sgt. (Law Enforcement)	(779) 970-0234
<b>PROJECT-RELATED TELEPHONE NUMBERS</b>	
EA Engineering, Science, and Technology, Inc., PBC (HV)	(410) 584-7000
Joe Vondruska, STR (EA)	O: (847) 607-6485/C: (847) 271-8412
Rob Marcase, Health and Safety (EA)	O: (410) 329-5192/C: (717) 586-9878
Michele Bailey, Human Resources (EA)	O: (410) 527-2481/C: (410) 790-3795
Jeff Boltz, WNR Director (EA)	O: (410) 329-5179/C: (410) 804-9230
Marty Sneen, Scientist, Project Manager (EA)	O: (847) 607-6484/C: (847) 372-6332
Ken Cummings, Scientist, Field Lead (EA)	O: (847) 607-6475/C: (847) 271-8406
Marisol Sepúlveda, Scientist, Fish Health (Purdue)	(765) 496-3428
Michael Chanov, Scientist, Bioassay Lead (EA)	410 329-5120
Julie Holscher, MPC Environmental	(618) 469-5336
Lisa Stewart, MPC – Safety Training	(618) 546-5111
Lenzi Ippolito, MPC Environmental	O: (618)-469-5553/C: (618) 553-0144
Lisa Stewart, MPC Orientation	618-546-5111
MPC Emergency	(618)-544-2121 ext. 5300
Contract Laboratory	Ken Hunt (317) 228-3120 at Pace Indianapolis (Kenneth.Hunt@pacelabs.com)
UPS	1-800-742-5877
Federal Express	6.1.1.1 (1-800-GO-FEDEX)



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**Appendix A**  
**Standard Operating Procedure for Surface Water**  
**Sampling**

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# **Study Plan for the Assessment of Deformity, Erosion, Lesion, and Tumor (DELT) Anomalies at Marathon Petroleum Company's Robinson Refinery**

*Prepared for*

Marathon Petroleum Company, LP  
400 S Marathon Avenue  
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*Prepared by*

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August 2022  
Version: FINAL  
EA Project No. 1604001

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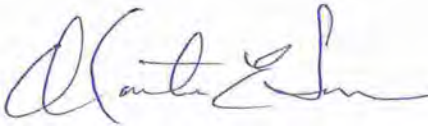
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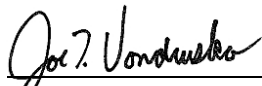


10 August 2022

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Marty Sneen  
Project Manager

Date



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Joe T. Vondruska  
Senior Technical Reviewer

10 August 2022

Date

August 2022  
Version: FINAL  
EA Project No. 1604001

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**LIST OF ACRONYMS AND ABBREVIATIONS**

°F	Degrees Fahrenheit
°C	Degrees Celsius
ANZI	American National Standards Institute
ASTM	American Society for Testing and Materials
ATHEL	Alternative Thermal Effluent Limitation
cat	Catalase
cDNA	Complimentary DNA
COVID-19	Coronavirus Disease 2019
CPE	Catch-Per-Unit-Effort
DC	Direct Current
DELT	Deformities, Erosion, Lesions/Ulcers, and Tumors
DISA	Defense Information Systems Agency
DNA	Deoxyribonucleic Acid
DO	Dissolved Oxygen
EA	EA Engineering, Science, and Technology, Inc., PBC
EDD	Electronic Data Deliverable
ELISA	Enzyme-Linked Immunosorbent Assay
EPA	Environmental Protection Agency
FDA	U.S. Food and Drug Administration
FHM	Fathead Minnow <i>Pimephales promelas</i>
FHI	Fish Health Index
FR	Fire Retardant
ft.	Feet
g	Gram(s)
g/L	Grams per Liter
GADPH	Glyceraldehyde 3 Phosphate Dehydrogenase
GPS	Global Positioning System
gst	Glutathione-s-Transferase
hr.	Hour(s)
lbs.	Pound(s)
ID	Identification
IDNR	Illinois Department of Natural Resources
IPCB	Illinois Pollution Control Board
MBI	Midwest Biodiversity Institute
mg/L	Milligrams per Liter



mL	Milliliters
mm	Millimeters
MPC	Marathon Petroleum Corporation
NBS	National Bureau of Standards
NFPA	National Fire Protection Board
oz.	Ounce(s)
PAH	Polynuclear Aromatic Hydrocarbons
PBI	Polybenzimidazole
pH	Potential Hydrogen
POTW	Publicly Owned Treatment Works
PPE	Personal Protective Equipment
QA/QC	Quality Assurance/Quality Control
qPCR	Quantitative Polymerase Chain Reaction
RNA	Ribonucleic Acid
RM	River Mile
sod	Superoxide Dismutase
TWIC	Transportation Worker Identification Card
µg/dL	Micogram per Deciliter
µL	Microliter
µS/cm	Microsiemens per centimeter
USEPA	United States Environmental Protection Agency
uv	Ultraviolet
vol	Volume
WBC	White Blood Cells

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## 1. INTRODUCTION

As set forth in Order and Opinion PBC 18-49 and in response to comments provided by the Illinois Department of Natural Resources (IDNR), the Illinois Pollution Control Board (IPCB) found that the record did not contain adequate information to determine if the synergistic effect of Marathon Petroleum Company's (MPC) Robinson Refinery thermal discharge and non-thermal stressors in Robinson Creek is causing an increased incidence of deformity, erosion, lesion, and tumor (DELT) anomalies on fish. Given that the proposed alternative thermal effluent limitations (ATELs) include a mixing zone without a zone of passage, the IPCB required as a condition to the ATELs that MPC conduct a study as suggested by the IDNR (PBC 18-49, 7 July 2020 IDNR Response, Attachment C). This study was designed to follow the IDNR recommended study with modifications to accommodate field implementation.

DELT anomalies are the group of anomalies for which a clear relationship has been established between their incidence (percentage) and water quality (Ohio EPA 1987). A high frequency of DELT anomalies is a good indication of a stress caused by sublethal stresses, intermittent stresses, and chemically contaminated substrates. The following is an overview of DELT anomalies and their causes in freshwater fishes:

- Deformities – These anomalies can include malformation of the head, spinal vertebrae, fins, barbels, and abdomen, and have a variety of causes including, but not limited to, toxic chemicals, heavy metals, viral and bacterial (e.g., *Mycobacterium*) infections, and parasites (e.g., *Myxobolus cerebralis*; Post 1983) (Ohio EPA 2015).
- Eroded fin, gill cover, barbel, or other body part – These are the result of chronic disease caused principally by flexibacteria invading the tissue and causing necrosis (Post 1983). Necrosis of the fins may also be caused by gryodactylids, a small trematode parasite (Ohio EPA 2015).
- Lesions and Ulcers – These appear as open sores or exposed tissue and can be caused by viral (e.g., *Lymphocystis*) and bacterial (e.g., *Flexibacter columnaris*, *Aeromonas*, *Vibrio*) infections (Ohio EPA 2015).
- Tumors – These result from the loss of carefully regulated cellular proliferative growth in tissue and are generally referred to as neoplasia (Post 1983). In wild fish populations, tumors can be the result of exposure to toxic chemicals. Baumann et al. (1987) identified polynuclear aromatic hydrocarbons (PAHs) as the cause of hepatic tumors in Brown Bullhead from the Black River (Ohio). Viral infections (e.g., *Lymphocystis*) can also cause tumors. Parasites (e.g., *Glugea anomala* and *Ceratonova shasta*; Post 1983) may cause tumor-like masses, but these are not counted as tumors. Parasite masses can be squeezed and broken between the thumb and forefinger, whereas true tumors are firm and not easily broken (Ohio EPA 2015).

This study consists of three elements: onsite thermal bioassay, field collections and DELTs assessment, and fish health assessment. The primary objective of this study was to determine whether the Robinson Refinery thermal discharge is causing an increased incidence of DELTs on

fish in Robinson Creek, particularly in Bigeye Chub and similar species. We hypothesized that Fathead Minnow exposed to the refinery effluent and Spottfin Shiner collected closest to the refinery effluent outflow would respond with decreased growth and lipid reserves; increased prevalence of DELTs and increased cortisol levels; altered blood cells differentials in fish showing DELT anomalies; and with a dysregulation in the expression of detoxification and oxidative stress genes compared to controls. We also hypothesized that increased water temperatures would exacerbate these changes and impact survival.

## 2. METHODS AND MATERIALS

### 2.1 ONSITE THERMAL BIOASSAY

The on-site thermal bioassay will consist of three site exposures and one control. The site exposures will consist of upstream, effluent, and downstream waters (Figure 1) conducted simultaneously with a dechlorinated tap water exposure (the control). The testing will be conducted at two temperatures to evaluate thermal stress. The studies will be conducted for 60 days or as long as the refinery remains within normal operating conditions. Study organisms will consist of adult (~6-month-old) male Fathead Minnows (*Pimephales promelas*). The two temperatures will represent a background cold water stream maximum condition and an elevated temperature condition that will mimic summer/fall variations.



**Figure 1. Aerial View of the Upstream, Effluent, and Downstream Exposure Water Collection Points**

USEPA states that when choosing test organisms, one should select a species that is representative of resident organisms, sensitive to site contaminants, relevant to the overall assessment endpoints, and consistent with data quality objectives (US EPA 1992). The test organisms should serve as surrogates for organisms present on the site. Based on this broadly

accepted framework, a commercially available minnow species, Fathead Minnow, which is in the same genus as Bluntnose Minnow (*Pimephales notatus*) will be used. Approximately six-month-old, sexually mature, male Fathead Minnows will be obtained from a scientific organism vendor (Aquatic BioSystems, Fort Collins, Colorado). Aquatic BioSystems is a full-service organism culturing facility specializing in the production and distribution of freshwater and marine organisms for aquatic toxicology, biomonitoring and other research activities. The organisms are completely laboratory reared using the latest information and technology available. This ensures the consistent production of organisms that are of the highest quality.

Test vessels will be of sufficient volume to not exceed the organism loading requirements. Loading will not exceed 7 grams/liter (g/L) in any chamber at test temperatures of 15°C and below. At 25°C, loading will not exceed 2.5 g/L at any time. In order to ensure applicable loading rates, testing will be conducted in 50-gallon plastic barrel troughs. Additionally, a 300-gallon reservoir of water will be recirculated through the testing chambers to increase the total water volume and decrease the total concentration of waste products. This water will be refreshed every other day. Each trough will contain 25-30 fish per tank. Three replicate tanks will be used for each test water. Fish will be randomly assigned to each test container.

The test vessels will be set-up in two separate trailers (Figures 2 and 3) with the same treatments (Control, Upstream, Effluent, and Downstream) but at different temperatures. In one environmentally controlled trailer, test vessels will be maintained at 20°C ±2°C while test vessels in the second environmentally controlled trailer will be maintained at 30°C ±2°C. Both trailers will have 16-hour light and 8-hour dark photoperiods with room temperatures monitored continuously.



**Figure 2. Aerial View of Routes to the Bioassay Trailers**



**Figure 3. Bioassay Trailers Footprint adjacent to the Wastewater Filter Press Building**

Water quality (temperature, pH, dissolved oxygen and conductivity) of the test solutions will be measured daily in one replicate per test concentration using a Star Orion A329 multimeter. Additionally, select parameters will be measured at three times throughout the study (i.e., beginning, approximate midpoint, and end). Based on the duration of testing, previous water chemistry results from the 316(a) demonstration (MBI 2017), and constituents commonly associated with DELTs (OEPA 1987), the following parameters were selected for analysis:

**Table 1. Water Chemistry Analytes to be Measured during the Thermal Bioassay Study**

1. Acenaphthene	44. Methylene chloride	87. Dieldrin
2. Acrolein	45. Bromoform	88. Chlordane
3. Acrylonitrile	46. Bromomethane	89. 4,4-DDT
4. Benzene	47. Chloromethane	90. 4,4-DDE
5. Benzidine	48. Dichlorobromomethane	91. 4,4-DDD
6. Carbon tetrachloride	49. Bromodichloromethane	92. Alpha-endosulfan
7. Chlorobenzene	50. Hexachloro-1,3,-butadiene	93. Beta-endosulfan
8. 1,2,4-trichlorobenzene	51. Hexachlorocyclopentadiene	94. Endosulfan sulfate
9. Hexachlorobenzene	52. Isophorone	95. Endrin
10. 1,2-dichloroethane	53. Naphthalene	96. Endrin aldehyde
11. 1,1,1-trichloroethane	54. Nitrobenzene	97. Heptachlor
12. Hexachloroethane	55. 2-nitrophenol	98. Heptachlor epoxide
13. 1,1-dichloroethane	56. 4-nitrophenol	99. Alpha-BHC
14. 1,1,2-trichloroethane	57. 2,4-dinitrophenol	100. Beta-BHC
15. 1,1,2,2-tetrachloroethane	58. N-nitrosodimethylamine	101. Gamma-BHC
16. Chloroethane	59. N-nitrosodiphenylamine	102. Delta-BHC
17. Bis(2-chloroethyl) ether	60. N-nitrosodi-n-propylamine	103. PCB-1242 (Arochlor 1242)

18. 2-chloroethyl vinyl ethers	61. Pentachlorophenol	104. PCB-1254 (Arochlor 1254)
19. 2-chloronaphthalene	62. Phenol	105. PCB-1221 (Arochlor 1221)
20. 2,4,6-trichlorophenol	63. Bis(2-ethylhexyl) phthalate	106. PCB-1232 (Arochlor 1232)
21. Chloroform	64. Butyl benzyl phthalate	107. PCB-1248 (Arochlor 1248)
22. 2-chlorophenol	65. Di-n-Butyl Phthalate	108. PCB-1260 (Arochlor 1260)
23. 1,2-dichlorobenzene	66. Di-n-octyl phthalate	109. PCB-1016 (Arochlor 1016)
24. 1,3-dichlorobenzene	67. Diethyl Phthalate	110. Toxaphene
25. 1,4-dichlorobenzene	68. Dimethyl phthalate	111. Antimony
26. 3,3-dichlorobenzidine	69. Benzo(a) anthracene	112. Arsenic
27. 1,1-dichloroethylene	70. Benzo(a) pyrene	
28. 1,2-trans-dichloroethylene	71. Benzo(b) fluoranthene	113. Beryllium
29. 2,4-dichlorophenol	72. Benzo(k) fluoranthene	114. Cadmium
30. 1,2-dichloropropane	73. Chrysene	115. Chromium
31. 1,3-cis-dichloropropene	74. Acenaphthylene	116. Copper
32. 1,3-trans-dichloropropene	75. Anthracene	117. Cyanide, Total
33. 2,4-dimethylphenol	76. Benzo(ghi) perylene	118. Lead
34. 4,6-dinitro-2-methylphenol	77. Fluorene	119. Mercury
35. 2,4-dinitrotoluene	78. Phenanthrene	120. Nickel
36. 2,6-dinitrotoluene	79. Dibenzo(a,h) anthracene	121. Selenium
37. 1,2-diphenylhydrazine	80. Indeno (1,2,3-cd) pyrene	122. Silver
38. Ethylbenzene	81. Pyrene	123. Thallium
39. Fluoranthene	82. Tetrachloroethylene	124. Zinc
40. 4-chlorophenyl phenyl ether	83. Toluene	125. 2,3,7,8-TCDD
41. 4-bromophenyl phenyl ether	84. Trichloroethylene	
42. Bis(2-chloroisopropyl) ether	85. Vinyl chloride	
43. Bis(2-chloroethoxy) methane	86. Aldrin	

For each of the three collection periods, eight (8) water samples (4 exposure waters x 2 temperatures) will be collected and analyzed for the aforementioned parameters. MPC will be responsible for contracting the laboratory, which includes obtaining the required sample bottles prior to each collection and shipment of collected samples to the laboratory. EA will be responsible for collecting the samples and documenting them on a chain-of-custody form. General guidance for Surface Water Sampling, Sample Preservation and Container Requirements, and Chain-of-Custody Forms are provided in Appendices A, B, and C, respectively. Sample identification nomenclature is provided in Table 2:

**Table 2. Water Chemistry Sample Identification Nomenclature**

Exposure Temperature	Exposure Water	Sample Identification
20°C	Control (CON)	20-CON-ddMMMyyyy <sup>(a)</sup>
20°C	Upstream (UPS)	20-UPS-ddMMMyyyy
20°C	Effluent (EFF)	20-EFF-ddMMMyyyy
20°C	Downstream (DNS)	20-DNS-ddMMMyyyy
30°C	Control (CON)	30-CON-ddMMMyyyy
30°C	Upstream (UPS)	30-UPS-ddMMMyyyy
30°C	Effluent (EFF)	30-EFF-ddMMMyyyy
30°C	Downstream (DNS)	30-DNS-ddMMMyyyy

(a) For example, 25JUL2022



Each test day, test organisms will be visually observed to record any mortalities and the presence or absence of DELT anomalies (Ohio EPA 2015). Dead organisms will be removed when observed and also examined for DELTs. If lesions are present, they will be swabbed and submitted for bacterial analysis.

Water for each of the three treatments (effluent, upstream, and downstream) and control will be exchanged every other day. Water from each of the locations will be collected using a dedicated stainless steel submersible pump. The water will be pumped into a 275-gallon plastic tote and transported to the testing trailer. The water will be allowed to acclimate to the test temperature for at least 1 hour prior to use. The water will be pumped from the tote to the reservoir tank in the testing trailers. Prior to the transfer, the reservoir tanks and accumulated waste from the exposure tanks will be pumped out to the facilities holding basin.

The system will be set-up as a modified flow through system, whereby the water will be recirculated through the tanks at a rate of approximately two volume addition per day. Flow rate will be documented weekly. The flow through system for each water source (i.e., Control, Upstream, Effluent, etc.) will be replenished every other day to minimize loss in water volume and water quality due to degradation, uptake, or evaporation.

In addition to the physical observations, fish health and stress will be assessed at beginning, midpoint, and end of the study. Details of the field and laboratory analysis for fish stress and health markers as well as lesion bacterial sample collection are provided in Section 2.3 and will be processed and analyzed in partnership with Dr. Maria Soledad (Marisol) Sepúlveda's laboratory at Purdue University.

## 2.2 FIELD COLLECTIONS IN ROBINSON CREEK

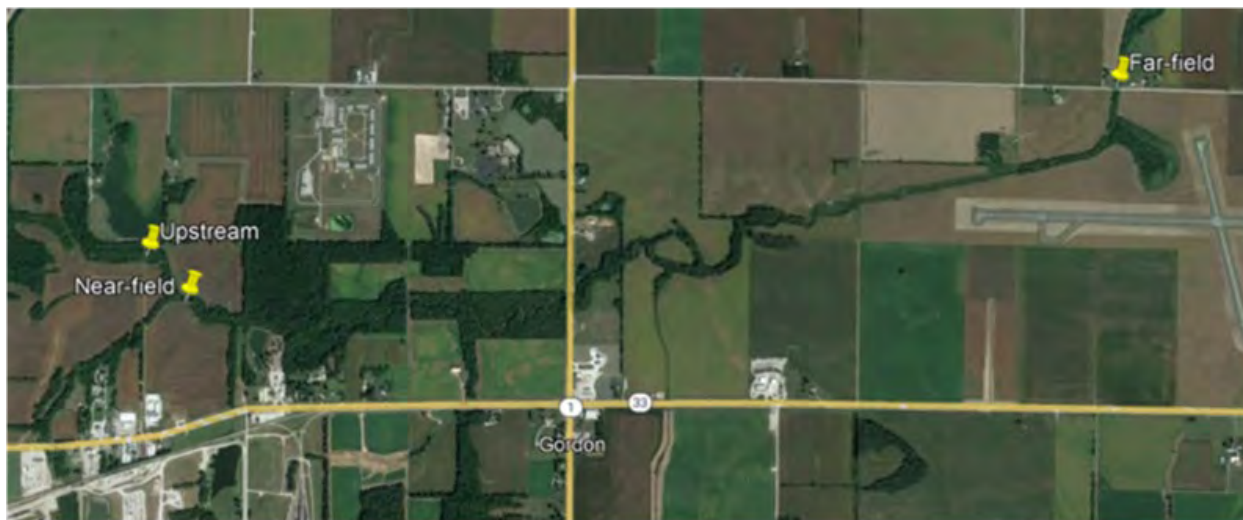
To evaluate fish health along a gradient both upstream and downstream of the discharge, three sampling zones will be established (Table 3 and Figure 4).

**Table 3. Descriptions of Robinson Creek Sampling Zones**

Zone	RM	Description
1	5.2	Ambient conditions, upstream of the MPC thermal discharge and downstream of the Robinson Publicly Owned Treatment Works (POTW). This zone will be near MBI's Location RC04 (MBI 2017).
2	5.0	Near-field zone beginning immediately downstream of the MPC thermal discharge. This zone will be near MBI's Location MPMZ (MBI 2017).
3	1.0-2.0	Far-field zone within the lower reaches of Robinson Creek. This zone will be near MBI's Location RC09 (MBI 2017).

An attempt will be made to establish zones like those sampled for the 316(a) demonstration; however, they will ultimately be configured based on the thermal discharge and available habitat at the time of sample collection. Sampling zones will be documented via a hand-held Global Position System (GPS). Fish surveys will be conducted mid-summer (August) and fall (late September or early October) 2022 with the mid-summer event conducted during the bioassay study, at or near the midpoint of the study. Sampling during summer and fall will capture those

seasons when water temperatures are warmest and stream flow is lowest, compared to other seasons, and therefore represent worst case conditions. EA has been issued Scientific Collection Permits and an Endangered and Threatened Species Permit by the IDNR that includes Robinson Creek (Appendix D).



**Figure 4. Aerial View of the Robinson Creek Field Study Sampling Zones**

### 2.2.1 Robinson Creek Fish Health Specimens

To minimize stress, seining will be conducted to collect the specimens that will be evaluated for stress and health markers. Depending on habitat, either a 30-ft bag seine with 1/8-inch Ace mesh or a 10-ft straight seine with 1/8-inch Ace mesh will be used. Sampling will be conducted for up to 90 minutes at each zone, depending on the number of target specimens collected. Based on 316(a) demonstration fish community data from Robinson Creek, target fish species will likely be Creek Chub (*Semotilus atromaculatus*), Silverjaw Minnow (*Ericymba buccata*), Spotfin Shiner (*Cyprinella spiloptera*), or Bluntnose Minnow (*Pimephales notatus*). Only adult male specimens will be retained for analyses. Details of the field and laboratory analysis for fish stress and health markers as well as lesion bacterial sample collection are provided in Section 2.3.

### 2.2.2 Robinson Creek DELT Anomaly Assessment

In order to characterize and compare the incidence of DELTs among the three sampling locations, a standardized 200-meter long zone will be electrofished at each location after the health and condition specimens have been collected. If specific conductance is below 2000  $\mu\text{S}/\text{cm}$  at a given location, electrofishing will be conducted using a longline or pram method. A Smith-Root 1.5 KVA control box will provide pulsed DC output powered by a 2,000-watt generator. If specific conductance is  $\geq 2000$   $\mu\text{S}/\text{cm}$ , a Smith-Root VVP-15 electrofisher will be utilized, powered by a 5,000-watt generator. In either case, one crew member will primarily operate an electrified probe while another will collect the stunned fish and monitor the electrofishing system. A barrier net (seine) will be deployed across the entire width of Robinson Creek immediately upstream of the MPC thermal discharge Outfall 001 prior to sampling to prevent fish movement between the upstream ambient zone and the downstream near-field zone.

All fish collected will be identified to species, counted, and examined for DELT anomalies. This information will be recorded on a project-specific fish sampling data sheet (Appendix E). The incidence of DELT anomalies will be recorded following procedures outlined by Ohio EPA (2015). Fish identifications will be made using *An Atlas of Illinois fishes: 150 Years of Change* (Metze et al. 2022), and scientific nomenclature will follow Metze et al. (2022) and Van der Laan et al. (2022).

No specimens collected by electrofishing will be analyzed for the health and condition bloodwork indices because electrofishing is very stressful to fish. However, all fish that display lesions will have the lesion swabbed to identify and quantify the local bacteria present.

In-situ water quality measurements of water temperature, DO, specific conductance, and pH will be collected at mid-depth at each sampling zone. Water clarity will be measured at each zone using a Secchi disk, depending on depth. These physicochemical measurements will also be recorded on the project-specific fish sampling data sheet (Appendix E). The suggested IDNR study indicates that water chemistry samples be collected during field sampling. Therefore, water chemistry samples will be collected for analysis at Day 0, Day 30, and Day 60 for the Control, Upstream, Downstream, and Effluent sources.

## **2.3 FISH HEALTH ASSESSMENT**

Randomly selected bioassay test fish and select species of field collected wild fish will be sampled to analyze blood stress and health markers that will consist of plasma cortisol, white blood cell counts, oxidative stress, and indices of nutrition. In addition, throughout the bioassay and during each field sampling trip, fish that display lesions will have the lesion swabbed to identify and quantify the local bacteria present.

### **2.3.1 Bleeding, Necropsy, and Sample Identification**

#### Recording of Holding Conditions:

- We will monitor water temperature and DO in the coolers used to transport the fish.
- A data logger which will record temperature every 5 min will be placed in every cooler used to transport the fish. Coolers will also be kept aerated using battery-operated air pumps.

#### Bleeding:

- Make sure you have all supplies needed for bleeding:
  - Pre-weighed MS222 and sodium bicarbonate powders for anesthesia and buckets to anesthetize fish
  - Measuring board
  - Digital scale (0.001 g and 2000 g)
  - Blades
  - Capillary tubes and clay
  - Pre-labeled 15 mL conical tubes for storing capillary tubes

- Syringes and needles
  - Green tops
  - Trash bags
  - Cooler with ice
  - Sharpies and pencils
  - Sharps box
- Put disposable gloves on.
  - In order to anesthetize the fish, empty contents from one of the vials containing powered tricaine methane sulfonate (MS-222) buffered with equal amounts of sodium bicarbonate and targeting a 150 mg/L concentration in the anesthesia container. Mix well.
  - For FHM, anesthetize fish, one at a time, in a 1 L plastic container filled halfway. For larger fish, use a 5-gal bucket filled  $\frac{3}{4}$  of the way.
  - Wait for ~ 5 minutes until the fish can't maintain equilibrium and opercular movement slows down. Net the fish out and pat excess water with paper towels.
  - Measure (Total Length in mm) with ruler.
  - Weigh whole fish in digital scale (in mg).
  - Place the fish on its side on the dissecting board. With one hand hold the fish head down and with the other make a single, quick incision in the peduncle area with a blade.
  - Have three capillary tubes ready to use to collect blood by capillary action. Fill tubes no more than ~ 90% of their capacity. Seal one end with clay. Place tubes inside a pre-labeled 15-mL conical tube. Place conical tube in cooler and keep on ice until centrifuging for collection of plasma.
  - If larger than 50 grams (g), fish will be bled from the caudal peduncle using 1 mL syringes fitted with 21 or 22 gauge 1 to 1  $\frac{1}{2}$ -inch needles. Place the fish on its side on the dissecting board and have a second person help hold the fish down.
  - Insert needle bevel up. If bleeding is unsuccessful, you can turn your fish around and try bleeding again from the other side or from the ventral area. Apply gentle suction with the syringe and collect approximately 1 mL of blood.
  - Make sure you use a new needle and a new syringe if you get any blood in it or it will clot.
  - Without the needle attached to the syringe, dispense blood into the heparinized plastic tube (green top). Label your tube with Fish ID # and treatment. These are not permanent labels, so you do not need to write all the info.
  - Mix gently for at least one minute!! Place in cooler with ice.
  - Dispose of all blades, syringes and needles in the sharps box.
  - Place capillary tubes in holding containers (in groups of 4) and spin in the field at 13,800  $\times$  g for 5 minutes.
  - Break the capillary tube at the plasma line and using a 1 mL syringe, blow out the plasma into a pre-labeled cryovial. Pool all the plasma from one fish into each tube.
  - Immediately place in a cooler with dry ice and store at -80°C at Purdue until processed for cortisol.

Necropsy:

- Make sure you have all supplies needed for necropsies:
  - Pre-weighed MS222 and sodium bicarbonate powders for euthanasia and buckets to euthanize fish
  - Dissecting tray and tools
  - Digital scale for weighing organs (0.001 g)
  - Pre-labeled tubes with RNA later for storing livers for gene expression
  - Empty pre-labeled tubes for storing livers for lipid content
  - Squirt bottle with 75% ETOH
  - Culture swabs
  - Paper towels
  - Trash bags
  - Paper envelopes for fish scale
  - Cooler with ice
  - Sharps box
- Put disposable gloves on.
- In order to euthanize the fish, empty contents from one of the vials containing powered tricaine methane sulfonate (MS-222) buffered with equal amounts of sodium bicarbonate and targeting a 500 mg/L concentration in the euthanasia bucket. Mix well.
- Place fish in euthanasia solution until no opercular movements are observed.
- Net the fish from the euthanasia bucket and place on a dissecting tray.
- Evaluate the external surface and note the general body condition of the fish, identify and note lesions on the skin, fins, and eyes.
- Using a bacterial culture swab, gently swab over erosions, ulcers, and areas that look abnormal (e.g., abnormal color, missing scales). Take note of where samples were collected.
- Label swab with Fish ID # and treatment. These are not permanent labels, so you do not need to write all the info.
- Place swab in cooler with ice.
- Proceed to open the abdominal cavity by cutting along the ventral midline from the gills to the anus. Remove the liver and split into two vials, one for lipid (which has no fixative) and one for gene expression which contains RNA later.
- Place sample in the cooler with ice.
- Place sample with no fixative in cooler with dry ice.
- Collect testes and weigh them.
- For wild fish, determine sex by macroscopically examining the gonads. If large enough, dissect and weigh.
- For wild fish, collect a dozen scales above and below the lateral line and save in paper envelopes.
- For FHM and small fish, place the remaining carcass in a pre-labeled Ziplock bag in cooler with dry ice. Store samples at  $-80^{\circ}\text{C}$  upon arrival at Purdue.

- For larger fish, collect fin samples (caudal, dorsal and pectoral) and place in pre-labeled bags in cooler with dry ice. Store samples at  $-80^{\circ}\text{C}$  upon arrival at Purdue.
- Discard fish remains in a garbage bag. Bring back to Purdue for proper disposal.
- Change gloves and clean dissecting board and tools with 75% ETOH in between fish.

Each sample will be assigned a unique alphanumeric identifier upon collection, SPP-#-LOCAL-ddMMM-FHI, where: SPP denotes the fish species (Table 4); # represents fish ID; LOCAL identifies where the species is collected (Table 4); ddMMM (e.g., 08AUG) denotes when the sample is collected; and FHI is the fish health index to be analyzed (Table 4).

**Table 4. Fish Health Index Sample Codes**

Species <sup>(a)</sup>	Locale	Fish Health Index
Fathead Minnow (FHM)	20°C Control (20CON)	Plasma Cortisol (COR)
Bluntnose Minnow (BNM)	20°C Upstream (20UPS)	White Blood Cell count (WBC)
Creek Chub (CCH)	20°C Effluent (20EFF)	Oxidative Stress (OXS)
Silverjaw Minnow (SJM)	20°C Downstream (20DNS)	Nutritional Condition (LIP)
Spotfin Shiner (SFS)	30°C Control (30CON)	Bacteria Culture (BAC)
	30°C Upstream (30UPS)	
	30°C Effluent (30EFF)	
	30°C Downstream (30DNS)	
	Zone 1 Robinson Creek (Z1ROB)	
	Zone 2 Robinson Creek (Z2ROB)	
	Zone 3 Robinson Creek (Z3ROB)	

(a) Additional species will be added, as necessary.

For example, if blood is drawn from the third Fathead Minnow processed on 15 August 2022 from the 30°C effluent exposure tanks, the sample identification code would be: FHM-3-30EFF-15AUG-COR.

## 2.3.2 Fish Health Indices

### 2.3.2.1 Plasma Cortisol

Once samples are received at the University of Purdue Laboratory, capillary tubes will be spun in a microhematocrit centrifuge at  $13,800 \times g$  for 15 minutes and plasma collected and stored in pre-labeled cryovials which will be flashed frozen in liquid nitrogen and stored at  $-80^{\circ}\text{C}$  at the Purdue laboratory until processed for cortisol analyses.

Cortisol levels will be quantified using a cortisol Enzyme-Linked Immunosorbent Assay (ELISA) kit according to the manufacturer's instructions. A kit sold by Salimetrics will be used, as it has worked for Zebrafish plasma with a sensitivity of less than  $0.007 \mu\text{g}/\text{dL}$  (Grzelak et al. 2017). The kit requires a total of  $25 \mu\text{L}$  plasma. However, samples will be run in duplicate using a plate reader located in the Aquatic Molecular Laboratory at Purdue University. Therefore, a total of  $50 \mu\text{L}$  of plasma will be necessary to run one sample in duplicate, which should not be an issue for fish greater than 50 g. For the bioassay Fathead Minnows in particular, plasma will be

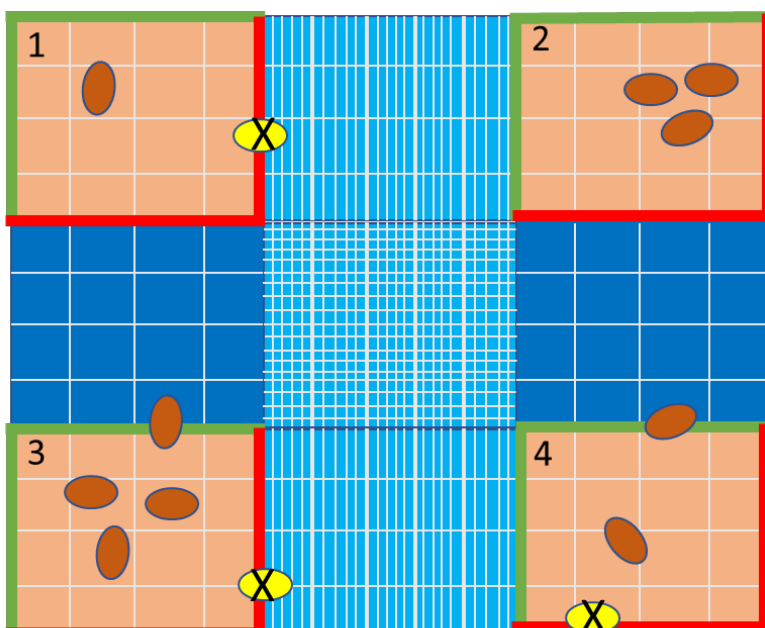
pooled from three individuals to obtain one capillary tube/sample (50  $\mu$ L whole blood total). Assuming that approximately 50% of each blood sample is made up of red blood cells, these three fish should provide enough plasma to run this assay and an additional approximately 20  $\mu$ L of blood to quantify white blood cells.

### 2.3.2.2 White Blood Cell Counts

#### Unopette Method:

The Unopette® uses a disposable diluting pipette system that provides a convenient, precise, and accurate method for obtaining a white blood cell (WBC) count. The diluent lyses the red blood cells but preserves the WBCs.

- **Clean the hemocytometer with 75% ETOH and dry with Kim wipes. Place coverslip on top.**
- **Fill up the small capillary tube that comes with the kit with whole blood and insert into vial with dye.**
- **Mix for a few seconds.**
- **Collect a sample using the larger capillary tube that comes with the kit and load it into the hemocytometer.**
- **Using a microscope, focus on the grid lines of the counting area with a 4-10x objective.**
- Out of the 9 squares, the 4 corner ones are used for WBC count (Figure 5).
- **Count the cells in one set of 16 squares (1 $\times$ 1 mm square area; the orange area). You should set a counting rule and begin counting at the top left (#1) and proceed through the 16 small squares to #2, #3 and #4.**
- Multiply the total number of cells counted by 50 and report in number of **WBCs/mm<sup>3</sup>**.



**Figure 5.** View of cell counting chambers in hemocytometer. For white blood cells, count only the four corners (orange). Only count cells that fall on the green line, not on the red line. Repeat counts if the difference between two replicates is  $> 15\%$ .

### **Blood Smears:**

Blood smears will also be made as a secondary method to quantify WBCs.

- a. Make two slides per fish. Using a pencil (not a pen which will rub off when fixing slide with methanol) label the slide on the frosted area. Use the labelling system already described.
- b. Using a capillary tube or a fine tip disposable pipette, place a small drop of blood at the edge of a clean microscope slide (slide A)
- c. Hold another clean slide (slide B or spreader slide) with thumb and index finger at  $45^\circ$  angle to slide A. Keeping the same angle, slowly move slide B toward the blood and contact these two slides with the blood. Spread the blood across to the edges of these two slides. Make sure that the  $45^\circ$  angle wedge shape constructed with two slides is filled with the blood before you spread slide B over slide A.
- d. Still holding slide B in a  $45^\circ$  angle, move the slide toward the end of slide A to make a thin even blood smear on slide A. This should be done in one movement without stop.
- e. Air dry slides and proceed to fix and stain using the kit provided. The kit consists of one fixative (methanol) and two different stains. Insert each slide into each solution 3 times. Tap slide on paper towel in between stains. Carefully wash off excess stain with water in the sink.



- f. Let slide dry and examine under the light microscope from 10 to 100X. Use immersion oil for a better examination of cells.
- g. For a WBC differential count, a total of 100 WBCs need to be counted and categorized into either lymphocytes or granulocytes (i.e., neutrophils, eosinophils & basophils).

### 2.3.2.3 Oxidative Stress

The expression of three key genes in oxidative stress will be measured: Superoxide dismutase (*sod*), catalase (*cat*) and glutathione-s-transferase (*gst*) as these have been reported as sensitive genes in response to oxidative stress in fish (Salnino et al. 2009). Expression of cytochrome P4501A (*cyp1a*) will also be quantified as an excellent biomarker of exposure to a wide range of pollutants. Gene expression will be quantified using standard qPCR protocols developed at Purdue University (e.g., Godfrey et al. 2017). Either GAPDH and/or beta-actin will be used as reference genes. If the expression of reference genes is too variable, additional genes will be selected and their combined expression may be used as the threshold to which results are compared. Primers for Fathead Minnow have been published for all of these genes (Bertucci et al. 2020).

### 2.3.2.4 Nutritional Condition

The quantity of total lipids in liver samples will be used to assess nutritional condition. A standard gravimetric method will be used as adopted for small liver (biopsy) samples of less than 100 mg, as described in Starke et al. (2010). An aliquot (2 mL) of a 3:2 mixture (vol/vol) of hexane and isopropanol will be added to each sample and after 24 hours at 20°C, the supernatant will be removed, weighed, and total lipid content determined gravimetrically as mg/g of liver.

### 2.3.2.5 Lesion Swabbing for Bacteria Culture

Using sterile swabs and avoiding cross contamination, lesions will be swabbed using sterile pipettes, which contain a culture media that preserves the microorganisms that could be present. Swabs will be kept on ice and the same day, plated on 2% blood agar plates and incubated at 20°C to 25°C for 2 to 3 days. Colonies will be counted and a subset saved for DNA analysis using a “shot-gun” genomics approach. A maximum of 60 samples will be cultured for the bioassay and field studies, respectively.

In order to characterize microbial taxonomic composition, a subset of microbial DNA directly extracted from plates will be submitted for sequencing using the 16S rRNA gene amplification method, targeting the V3/V4 region of the gene. Amplification products will be sequenced on an Illumina MiSeq platform (300 bp paired-end raw reads) either at Purdue’s Genomics Core or at a commercial lab.

- a. Lesions will be cultured on two different media (MacConkey and Levine Eosin Methylene Blue) to provide ideal culture conditions for a wide range of bacteria.
- b. Working under the biosafety cabinet in FORS 119, inoculate culture plates with swab samples.

- c. Hold the swab in one hand and lift the lid of the culture petri dish with the other. Use the lid of the petri dish to protect the agar from aerial contamination.
- d. Drag the swab in a zig-zag pattern until all surface of the plate is covered,
- e. Place lid back on petri dish and using sharpie, label the bottom of plate with your initials, date and time.
- f. Incubate plates in an inverted position at 25°C for 120 hours.
- g. Check for colony development every 24 h. Count colonies.
- h. Collect samples for DNA sequencing from a representative number of colonies and store in pre-labeled cryovials in the -80°C freezer.

### 2.3.2.6 Fish Health Assessment Sample Summary

Bioassay test specimens will be collected for analysis at the beginning, midpoint, and end of the study. Since the specimens collected at the beginning of the 60-Day study were collected prior to exposure to any treatments, five specimens from each treatment were collected. For the midpoint and end of study collections, cortisol will be sampled in five replicates for each water type and temperature exposure (40 samples per sampling period, 120 samples for the midpoint and end periods). Additionally, three types of blood and tissue samples will be collected in five replicates for each water type and temperature exposure (40 samples per sampling period, 120 samples for the midpoint and end periods). These samples will consist of white blood cell counts (leukocytes) to quantify infection levels and immune function, tissue samples to quantify oxidative stress, and liver samples to assess nutritional condition using total lipids.

As described above, field collections for health and stress markers will be conducted by seining during summer and fall. For each location and sampling event, fish health and condition will be assessed by collecting 10 replicate plasma cortisol samples (up to three fish per sample) and an additional 10 replicate fish samples (one fish per sample) for white blood cell counts, oxidative stress, and nutritional condition. In addition, fish health specimens collected by seining and all fish collected electrofishing will be examined for DELTs. Fish that display lesions will have the lesions swabbed to identify and quantify the local bacteria present. Although the number of fish that exhibit lesions is unknown, we estimate that bacterial swabs could be collected from up to 60 bioassay test fish and 60 wild caught fish. Table 5 summarizes the number and types of samples that will be laboratory analyzed.

**Table 5. Summary of the Number of Samples and Fish to be Analyzed for the Fish Health Assessment Indices**

Study Type	Response	Number of Samples Needed/Estimated*	Estimated Number of Fish Needed	Design
Bioassay	Plasma cortisol	120	360	2 temperatures x 3 sampling periods x 4 water types x 5 replicate samples
	White blood cell counts	120	120	
	Liver oxidative stress	120		
	Liver total lipids	120		
	Bacterial swabs*	60*	60	All fish with lesions

Field	Plasma cortisol	60	90	3 field zones x 2 sampling events x 1 species x 10 replicate samples
	White blood cell numbers	60	60	
	Liver oxidative stress	60		
	Liver total lipids	60		
	Bacteria swabs*	60*	60	All fish with lesions
<b>Total</b>		<b>840</b>	<b>750</b>	

### **3. DATA ANALYSIS AND REPORTING**

#### **3.1 ONSITE THERMAL BIOASSAY**

Tabular and/or graphical summaries will be provided in the report for:

- Water chemistry results from each of the three collection periods, including comparisons with results from the 316(a) study and Illinois General Use Water Quality Standards at 35 Ill. Adm. Code 302, Subpart B;
- Water temperature, DO, pH, and specific conductance of each test vessel in terms of daily minimum, mean, and maximum values;
- Daily test organism observations from each test vessel in terms of the presence or absence of DELTs and the number of dead organisms; and
- Fish swabbed and submitted for bacterial analysis.

Narrative descriptions of these results will be presented in the report along with a detailed methods section that documents all quality assurance and quality control procedures utilized (see Section 4). Any deviations or nonconformances will be documented and discussed with respect to their impact on the thermal bioassay results.

#### **3.2 ROBINSON CREEK DELT ANOMALIES ASSESSMENT**

Field data will be entered into a Microsoft Excel spreadsheet and then exported into a SAS (Version 9.2) database. Electrofishing data will be reported as number, catch-per-unit-effort (CPE, number per 300 m), and percent abundance for each species segregated by sampling zone and sampling period. DELT anomaly data will be presented as the number and percent afflicted by species and for species combined and compared spatially and temporally. The DELT anomaly data will also be compared to the 2016 results as appropriate. These results will be discussed in the final report, which will also include a detailed description of the sampling methodologies along with quality assurance and control procedures (see Section 4). The raw data will be included in an appendix.

#### **3.3 FISH HEALTH ASSESSMENT**

Tabular and/or graphical and statistical summaries will be provided in the report for (sample sizes provided under Table 5):

- Fish body sizes (weight and total length);
- Plasma cortisol concentrations;
- Total number of white blood cells;
- Expression of hepatic genes related to oxidative stress; and
- Total lipids in liver.

All raw data will be provided in the form of Excel files and analyzed using R (2022.07.01). Summary statistics will include mean, standard error of the mean, and ranges. For the bioassays, means will be compared between treatments and controls for each time point. Means of feral fish will be compared across sites for each of the time points. In the final report, a table with all the statistical results, including  $p$  and  $F$  values, will be provided for each statistical analysis performed.

## 4. QUALITY CONTROL

This study plan provides EA and Purdue University staff (the project team) with guidance regarding sampling methodologies, the equipment and supplies required, specifications regarding acceptable calibration intervals and procedures for various field and laboratory equipment, communication, quality control and assurance measures, and health and safety requirements. It also establishes various other protocols to be followed throughout this project.

In accordance with EA's Corporate Quality Management Plan (the master document for all disciplines at EA), study plans efficiently and effectively promote quality through consistency. This study plan is a project-specific document that integrates the methodologies and guidance with detailed specifications. It ensures that the study objectives will be met and that the integrity of the project team will be maintained. It also allows each staff member to understand his or her duties and responsibilities.

### 4.1 EQUIPMENT CALIBRATION

#### 4.1.1 Measuring Boards

Measuring boards and rulers used to determine lengths of fish are calibrated once after purchase, manufacture, or repair. The measuring board is calibrated with a ruler or tape that is certified by the manufacturer to be traceable to the National Bureau of Standards (NBS). Ten randomly selected points between 20 and 600 mm are visually checked against the standard ruler. Only those measuring boards that are within the stated accuracy of the standard ruler ( $\pm 1.5$  mm) are used. Measuring boards that are outside the accuracy of the standard ruler are discarded. The results of the calibration are entered on a Calibration Record Form that resides in an equipment-specific file folder.

#### 4.1.2 Measuring Scales

Spring scales are calibrated semi-annually by weighing 3-5 weights that are within the appropriate weight range. All weights are class T or better and are certified by the manufacturer to be traceable to the NBS. The readings are compared to the known weight and the results are recorded on a Calibration Record Form that resides in an equipment-specific file folder. Scales having less than 10 percent error are retained in service, whereas those that exceed 10 percent error are adjusted and recalibrated or removed from service and destroyed.

#### 4.1.3 Bioassay Temperature Monitoring System

The temperature monitoring system will be purchased from the Ideal Sciences and therefore tested and calibrated prior to the start of the study. It will be cross-checked daily against thermometer of the multi-probe water quality monitoring instrument used to measure DO, pH, and conductivity. These cross-checks will be documented on an Equipment Calibration Log (Attachment A in Appendix F, or equivalent).

#### 4.1.4 Water Quality Meters

Multi-probe water quality monitoring instruments will be used to measure DO, pH, and specific conductance. They will also be used to measure water temperature in Robinson Creek. The DO, pH, and specific conductance probes will be calibrated prior to coming onsite and once daily while onsite (Appendix F). The thermistors do not require calibration but will be cross-checked against a calibrated or reference thermistor at the same frequency as the other probes. The calibration standards will consist of:

- specific conductance: 1,000  $\mu\text{S}/\text{cm}$  conductivity standard;
- pH: buffer solutions of pH 7 and pH 10; and
- DO: in water-saturated air (or checked against a Winkler Titration).

The calibrations and cross-checks will be recorded on an Equipment Calibration Log or a Record of Calibration/Checking Form (Attachments A and B in Appendix F, respectively, or equivalent). The *YSI Professional Plus Calibrations Tips* document has been incorporated as Attachment C of Appendix F.

#### 4.1.5 Centrifuges

Centrifuges will be calibrated using manufacturers' instructions. Centrifuges will always be balanced to minimize vibrations.

### 4.2 FIELD SAMPLING

All project team members will be expected to have read and have on hand at all times a copy of the study plan. Experienced (30 years or more) project team scientists will conduct the Robinson Creek field surveys and be onsite to collect all fish health assessment samples, which will ensure strict adherence to the study plan, proper identification of fish captured, and sound judgment regarding the sample collection, preservation, processing, packing, shipping, and transportation procedures.

When collecting and handling fish, in order to preserve the integrity of the samples, we will maintain stress to a minimum, whether fish are collected via seining or electrofishing. When electrofishing, we will measure the water conductivity in the stream before electroshocking and adjust the electrofisher settings as needed to increase performance while at the same time, decrease potential damage to the fish. Because some of the responses we are measuring (i.e., cortisol and gene expression) can change quickly, we will process (anesthetize) fish as fast as possible after collection. The same care will be taken for laboratory fish so that fish are anesthetized as soon as collected from tanks. We will ensure all samples are properly labeled using a unique alphanumeric code that we will create for this project.

### **4.3 LABORATORY ANALYSES**

#### **4.3.1 Water Chemistry**

A laboratory will be contracted by MPC to measure the analytes listed in Table 1 for samples collected at the beginning, mid-point, and end of the DELTs bioassay. Analysis procedures will be conducted according to the contracted laboratories' Standard Operating Procedures, which will be documented in the laboratory reports.

#### **4.3.2 Fish Health**

For all tests described below, we will ensure all glassware used is properly cleaned and that pipettes and scales are properly calibrated as already described. Research grade chemicals will be used and purchased only from suppliers who guarantee purity. Any chemicals used for this work will be dated upon arrival and properly disposed of by expiration time. In general, samples will be run in duplicate and if differences between technical replicates is > 15%, samples will be re-run and a third replicate added.

#### **4.3.3 ELISA Tests for Quantifying Plasma Cortisol**

Our microtiter plate (Bio Tek Synergy HTX) reader is periodically controlled and maintained according to the specific recommendation of the suppliers. We will follow the QA/QC instructions provided by the vendor of the kits we plan to use (Salimetrics). Each plate will be run with a cortisol standard curve consisting of 6 concentrations plus two positive cortisol samples. Standard curves with  $R^2$  values of > 0.95 will be considered acceptable.

#### **4.3.4 Quantitative Polymerase Chain Reaction (qPCR)**

Our qPCR machine (CFX Connect Real-Time PCR) is periodically controlled and maintained according to specific recommendation of the suppliers. We will follow standardized protocols for RNA extraction, cDNA amplification, amplicon detection and confirmation, and use of positive and negative controls (US EPA 2004). We will ensure expression of reference genes is not significantly impacted by treatment/site of collection.

#### **4.3.5 Quantification of White Blood Cells**

Our microscope (Nikon Eclipse Ni scope with DS-Ri2 camera) is periodically controlled and maintained according to specific recommendation of the suppliers. If imaging is required, we will make sure scales are properly calibrated.

#### **4.3.6 Lipid Quantification in Livers**

We will run positive and negative controls during each batch of samples.



#### **4.4 FIELD AND LABORATORY DATA**

The project team will compare all (i.e., 100%) manually-entered field data against the hard copy field or laboratory data sheets. These comparisons will be kept as part of the project file, documented on a data processing log sheet (Table 6), and be made available to the client at their request. In addition, the comparisons will be done by an experienced scientist; data will not be checked by a non-scientist.

EA will perform a data assessment screening of the water chemistry analytical data packages and electronic data deliverables (EDDs) provided by MPC's contract laboratory. Data will be reviewed for completeness by comparing them to the chain of custody forms. Review of data usability will be accomplished by comparing the contents of the analytical data packages and QA/QC results to the requirements contained in MPC's Quality Assurance Plan and the respective analytical methods. EA will notify MPC of any deficiencies and work with the laboratory to resolve them.

We will ensure we maintain chain of custody forms for all the collections of biological samples. We will also ensure biological samples are maintained under the correct environmental conditions while in transit to the laboratories at Purdue University. A dataset will be considered final after cross-checking with field and lab-controlled forms and notebooks if needed.

**Table 6. Data Processing Log Sheet**

<i>EA ENGINEERING, SCIENCE, AND TECHNOLOGY, INC., PBC</i>			
<i>DATA PROCESSING LOG SHEET</i>			
<i>PROJECT NAME :</i> _____	<i>PROJECT NO. :</i> _____		
<i>DATA DESCRIPTION :</i> _____	<i>SAMPLE PERIOD :</i> _____		
<i>DATE</i>	<i>INITIALS</i>	<i>FILE NAME</i>	<i>ACTIVITY ( digacode, enter, proof, double check, etc.)</i>
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
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_____	_____	_____	_____
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## 5. HEALTH AND SAFETY

Since safety is of the utmost importance, no personnel will be required or instructed to work in surroundings or under conditions that are unsafe or dangerous to his or her health. Each individual team member will be responsible for complying with applicable safety requirements, wearing prescribed safety equipment, and preventing avoidable accidents.

### 5.1 ROBINSON REFINERY SITE-SPECIFIC REQUIREMENTS

The following information was obtained from the [Robinson Refinery Contractor](#) website and will be updated after onsite orientation. MPC Safety Procedure #12 *GENERAL SAFETY RULES* is provided in Appendix G.

#### 5.1.1 Prerequisites

Project team members must be enrolled in and tested by an MPC-approved Drug and Alcohol Test program, which is administered by DISA Global Solutions. They must also pass a DISA background security check and obtain a Transportation Worker Identification Credential (TWIC) card from the Transportation Security Administration.

#### 5.1.2 Site-specific Training

Project staff must take and successfully complete the Refinery-approved site-specific training prior to obtaining a Contractor Badge. Onsite training occurs in the Robinson Refinery Security Operations Center located directly east of the Refinery's Main Office Building, use the Route 33 entrance at the Wal-Mart stop light to access the Security Operations Center. Orientation is offered Monday through Friday at 7:30 am, 9:00 am, 10:15 am, 11:30 am, 1:00 pm, and 2:30 pm (central time zone). Contact Lisa Stewart to register for orientation, 618-546-5111 or [LStewart@Marathonpetroleum.com](mailto:LStewart@Marathonpetroleum.com).

Staff will obtain their refinery access ID card at the Refinery Security & Badging office located at: 400 S. Marathon Ave., Robinson, IL 62454. The following is required and will be verified by Refinery Security prior to issuing the Refinery access ID card:

- drug and alcohol testing compliance;
- background check;
- successful completion of the site-specific training; and
- a valid picture ID to receive your Refinery access ID card.

#### 5.1.3 Policies

##### 5.1.3.1 Smoking

Smoking (both regular and electronic) is permitted inside designated areas only. Smoking (both regular and electronic) is prohibited in vehicles within the refinery fence and in all MPC vehicles

at all times (Appendix G).

#### **5.1.3.2 Drug and Alcohol**

The possession of alcohol in unsealed or open containers as well as possession of unauthorized drugs on refinery property are prohibited. Closed/sealed containers of alcohol anywhere *other than* company parking lots outside the refinery fence line are also prohibited. No one under the influence of alcohol or illegal drugs is permitted in the refinery. Staff are responsible to notify the Medical Department in writing when they are taking prescription or nonprescription medicine or substance, which may impair their judgment or performance.

#### **5.1.3.3 Weapons**

Weapons and unauthorized firearms are prohibited on refinery property.

#### **5.1.3.4 Facial Hair**

Beards are prohibited within the refinery. However, EA has obtained a temporary waiver of the Marathon Petroleum Company LP Facial Hair Policy (Figure 5).

#### **5.1.3.5 Material Lifting**

When lifting objects >55 lbs. you should utilize one of the following options: 1) use two or more people to lift the load, or 2) use mechanical means of lifting (forklift, pallet jack, hand truck, etc.

#### **5.1.3.6 Spotter Usage Requirements for Vehicles**

Prior to entering process units, ensure provisions (spotters, barricades, etc.) are in place to prevent contact of the vehicle with process equipment. Consideration shall be given if a spotter will be required on roads not normally open to traffic, construction sites, or in heavily congested areas.

#### **5.1.3.7 Electronic Devices**

There are three types of Electronic Devices covered under #12 General Safety Rules:

- i. Type I – MPC Owned or Approved Devices with an MPC Approved Rugged Case.
- ii. Type II – Approved Contractor Devices with a case that meets all minimum requirements listed below & has an MPC Refining Approval Sticker obtained from the Safety Supervisor.
- iii. Type III – Personal Devices/Cell Phones.

The bioassay trailers are located within a restricted area. Therefore, project staff will use personal devices/cell phones only when inside the trailers or inside a vehicle. Cell Phone use in vehicles is limited to passengers, or when drivers are pulled over and parked at a complete stop or using a hands-free device. Cell Phones may not be used or on your person while operating a crane, man-lift, or anything similar in nature.

**Figure 5. Waiver of Facial Hair Policy**

Marathon Petroleum Company LP			
<b>Safety Procedure #21 RESPIRATORY PROTECTION</b>	Document No. 1021	Page Page 12 of 12	Revision No. 14
	Original Issue Date 02-82	Revision Date 12-6-2017	Next Revision Date 12-6-2021
	Document Custodian: Safety Control Group: REF (08) and 11-1014 - State Work Practices Revised: 2021.08.04 revised, incorporated in no longer revised		

Appendix C

MARATHON PETROLEUM COMPANY LP  
Ethane Refining Division

Date: 7/14/2022

WAIVER OF FACIAL HAIR POLICY

A temporary waiver of the Marathon Petroleum Company LP Facial Hair Policy is being temporarily issued to

EA Engineering (name employee and company, or company only) for

the purposes of performing the following work at the specified location within the refinery:

EA Engineering personnel will be working on site in temporary trailers set up west of the Ethane WWTP Miller process building. EA Engineering personnel will be performing a scientific study required by the refinery H2S-H2 Permit on full, untemperatures-controlled tower that with WWTP effluent and water from Robinson Creek.

This waiver is valid for the following dates: July 2022 - October 2022

*Kathy Harrison*  
Department Manager  
Ethane Refining Division

NOTES:

- This waiver is not required for delivery personnel and visitors covered under a visitor pass, as long as they are not required to wear respiratory protection.
- This waiver must be kept with the individual to which it was issued and a copy provided to Security when entering the gate.
- The waiver can only be written up to a 12 month period.

ALL INFORMATION CONTAINED HEREIN IS UNCLASSIFIED EXCEPT WHERE SHOWN OTHERWISE. THIS COPY IS CONTROLLED BY THE COMPANY AND IS NOT TO BE REPRODUCED OR TRANSMITTED IN ANY FORM OR BY ANY MEANS, ELECTRONIC OR MECHANICAL, INCLUDING PHOTOCOPYING, RECORDING, OR BY ANY INFORMATION STORAGE AND RETRIEVAL SYSTEM.

Marathon Petroleum Company LP			
<b>Safety Procedure #21 RESPIRATORY PROTECTION</b>	Document No.	Page	Revision No.
	1021	Page 12 of 12	14
	Original Issue Date	Revision Date	Next Revision Date
	02-02	12-6-2017	12-6-2021
Control Category: REF. 08 03 01 01 - Ref - Safe Work Practices Release: 2.00.1.001 revised, separate EHS register created			
Document Custodian: Safety			

Appendix C

MARATHON PETROLEUM COMPANY LP  
Ethanol Refining Division

Date: 7/14/2022

WAIVER OF FACIAL HAIR POLICY

A temporary waiver of the Marathon Petroleum Company LP Facial Hair Policy is being temporarily issued to EA Engineering (name employee and company, or company only) for the purposes of performing the following work at the specified location with the urgency:

EA Engineering personnel will be working on site in temporary trailers set up west of the Plant #1 WWTMP (the process building). EA Engineering personnel will be performing a scientific study required by the refinery. H2S Permit will be in place. Temperature control tank located with WWTMP effluent and water from Robinson Creek.

This waiver is valid for the following dates: JUN 2022 - October 2022

*Kathy Wilkinson*  
Department Manager  
Ethanol Refining Division

NOTES:

- This waiver is not required for delivery personnel and visitors covered under a visitor pass, as long as they are not required to wear respiratory protection.
- This waiver must be kept with the individual to which it was issued and a copy provided to Safety when entering the gate.
- The waiver can only be written up to a 12 month period.

ALLISTON: Printed copies should be sent with 020526. The user of this document is responsible for ensuring that the information contained herein is accurate and up-to-date. This report is printed on July 14, 2022 3:04:56 PM EDT.

### **5.1.4 Personal Protective Equipment**

Personal protective equipment and safety devices must be used as required and must not be altered in any manner. The use of damaged or malfunctioning personal protective equipment is prohibited.

#### **5.1.4.1 Safety Glasses with Approved Side Shields (ANSI - Z87.1)**

ANSI approved safety glasses with side shields must be worn at all times within the refinery where work is being performed. This includes maintenance shop areas, the laboratory, and at designated work sites away from the refinery.

Safety glasses with side shields are not required to be worn in the following locations:

- 1) West of 2 ½ Street,
- 2) Lunch/break rooms, control rooms, or plant offices, and
- 3) Inside vehicles with enclosed cabs (windows closed).

Contact lenses may be worn in conjunction with safety glasses/side shields. Workers who wear contact lenses should inform the refinery nurse of their use. The nurse will issue hard hat stickers indicating contact use.

#### **5.1.4.2 Goggles and Face Shields**

Employees are required to have ANSI Z87.1 approved chemical splash goggles on their person (i.e., on their hard hat, in a pouch on their belt, etc.) when in process areas, the tank farm, or designated off site locations where the potential for flying debris or chemical exposure exists.

At a minimum, unless engineering controls are in place, a face shield OR goggles must be worn when disconnecting hoses when potential for pressure exists.

Goggles must be worn for the following jobs or where there is risk of debris falling into the head/face area as a result of the work:

- 1) Handling powdered, granulated or dusty materials and loose insulation. Note that if there is the need to use a dust mask or half mask particulate respirator, goggles still must also be used.
- 2) Catching hydrocarbon samples.
- 3) Using pressurized air, steam, etc. to clean equipment.
- 4) Opening or transferring chemical totes via hoses.
- 5) When performing any internal cleaning of dirt/debris in vessels, tanks, exchanger shells, furnaces, etc.

A face shield (over safety glasses) must be worn for the following jobs:



- 1) When a flying chip hazard exists (i.e., grinding, chipping such as concrete/refractory, cutting, buffing, blasting, etc.),
- 2) While grinding or buffing vessels or equipment.
- 3) When using a torch/wand to light burners on heaters or boilers.
- 4) Operating an air powered nut gun/impact wrench.
- 5) When handling/working with hot products 140° F (molten sulfur, hot residue, hot condensate/boiler feedwater, etc.).
- 6) Operating a string trimmer during lawn maintenance.
- 7) When looking into fired heaters and boilers.

A face shield AND goggles must be worn for the following jobs:

- 1) Connecting/disconnecting lines or hoses in acid or caustic service.
- 2) When catching samples in acid or caustic service.
- 3) Cleaning, draining or repairing equipment which has been in acid or caustic service and not neutralized.
- 4) Loading or unloading of acids or caustics.
- 5) Initial line breaking or opening of equipment when potential for pressure exists.
- 6) Open sampling of liquids/products above 140 degrees F (non-engineering sample systems).

#### **5.1.4.3 Safety Toe Shoes (ASTM F2413)**

ASTM approved safety toe shoes with at least a ¼” defined heel must be worn at all times within the refinery property and at designated work sites away from the refinery when work is being performed.

ASTM approved shoes are not required to be worn in the following locations:

- Lunch/break rooms, control rooms, plant offices,
- Inside vehicles,
- Employees reporting to work or leaving work provided they go directly to their work area,
- Walking directly to or from personal vehicles or offices outside process unit battery limits,
- Truck drivers and vendors making deliveries or pickups of supplies, and
- Laboratory – shoes must be made of leather, rubber, or other non-absorbing material.

#### 5.1.4.4 Head Protection (ANSI Z89.1 Type 1 Class “E”)

All employees are required to wear an ANSI Z89.1 Type 1 Class “E” approved hard hat when in process areas, tank farm, designated off site locations where work is being performed, or new construction areas.

- 1) Hard hats must be changed at a minimum of every five years from the born-on date or when damaged or showing visible signs of wear (i.e., cracks, disfigurement, UV damage, etc.).
- 2) Hard hat suspensions must be changed at least annually.
- 3) Hair length longer than the shoulders must be kept under a hardhat when working around rotating equipment.

#### 5.1.4.5 Flame Resistant (FR) Protective Clothing

These procedures must be adhered to in order to provide adequate protection for workers in areas where there are recognized fire hazards and a reasonable probability that FR could mitigate burn injuries.

1. All FR clothing base garments (shirt/pant combo and/or coveralls) shall either be inherently FR material (e.g., Nomex, PBI) or FR treated cotton and cotton blends that are certified by an independent testing agency meeting NFPA 2112.
2. Seasonal accessories (e.g., UV face masks, cold weather beanies, or hard hat liners) shall also be meet NFPA 2112. (RSP Compliance Date - January 1, 2020)
3. Garments worn underneath base layers for warmth/cooling shall be made of natural fibers such as cotton, wool, or silk. This requirement does not include underwear.

**IMPORTANT:** Base layers made from synthetic materials such as polyester (e.g., Under Armor) are Prohibited.

4. FR shirts (not including outer FR garments (e.g., coats and sweatshirts with or without hoods, etc.) shall be tucked in, buttoned up, and sleeves rolled down when in FR required areas to comply with NFPA 2113.
5. Outer FR garments (e.g., coats, bibs, and sweatshirts with or without hoods, etc.) shall be made of FR fabric and adhere to NFPA 2112 requirements that are certified by an independent testing agency.
6. NFPA 2112 daily FR work wear garments shall be worn at all times under all outer FR garments.
7. Hole watch/Fire watch vests shall comply with ASTM D6413 Flame Resistant requirements. FR Rain Wear: (RSP Compliance Date - January 1, 2020)
8. All rain wear shall comply with ASTM D6413 Flame Resistant requirements, and shall be tested and comply in accordance with:
  - a. ASTM F2733 for flash fire, and
  - b. ASTM F1891 when the risk potential of an arc flash hazard exists.

**FR Disposable Coveralls:**

9. Disposable coveralls shall be made of FR fabric and are not required to meet NFPA 112 requirements.
10. Disposable coveralls shall comply with ASTM D6413.
11. Disposable coveralls shall comply with NFPA 2113 as it pertains to the care and maintenance during use.

NOTE: Any garments soiled with hydrocarbons or visibly tattered during work activities must be removed from service and replaced.

Each employee shall be responsible for the inspection and integrity of fire-resistant garments issued to them. Employees shall routinely inspect the garments for rips, tears, holes, discoloration, function of buttons, zippers, and fabric thinning due to age and repeated washings. Damaged clothing should be repaired or replaced.

FR shall be worn by all personnel in the refinery with the following exceptions:

- 1) Employees will be allowed entry into the refinery while wearing dresses, sleeveless shirts, & short pants, west of 2nd Street and including the E&I Shop, Main Warehouse, or while riding in an enclosed vehicle to Complex / PDU / Lab break rooms.
- 2) Employees reporting to work and leaving work, provided they go directly to their work area.
- 3) In Control Rooms and offices that are outside process unit battery limits.
- 4) Inside the Warehouses, E & I Shop, Machine Shop, Welding Shop, the Garage and Firehouses provided that no threat of flash fire exist.
- 5) While in the offices, main hallways and lunch/break rooms in the Laboratory.
- 6) In new construction areas that are not in an operating unit.
- 7) On refinery roadways.

**5.1.4.6 Hand Protection**

Gloves must be worn for jobs that have the potential for hand injury. Each person when in process areas, the tank farm, or designated off site locations where the potential for hand injury exists who is required to wear fire resistant clothing shall at least have general duty work gloves conforming to ANSI/ISEA 105 Level 3 at least in the palm, fingers and thumb of the glove for general operations and maintenance work.

For tasks with the potential of impact hazards, gloves with impact protection to the back of the hand and full length of the fingers are to be worn. (e.g., work with hammers, picking up blinds/valves, hand wrenching, flange bolts, impact gun tasks, tasks where hands and fingers can be pinched between the tool and a fixed object or material)

#### 5.1.4.7 Hearing Protection

Hearing protection is required to be worn inside the operating boundary (perimeter) of all process units, including during shutdown/turnaround periods. High noise areas in the plant may be designated by a yellow stripe and/or signs stating “Caution - Ear Protection Must Be Worn In This Area”. High noise areas are also encountered around operating equipment such as vacuum trucks, compressors and operating pumps in the tank farm. Hearing protection must be worn regardless of the time spent in these areas.

#### 5.1.4.8 Life Jackets

U.S. Coast Guard-approved life jackets must be worn at all times whenever there is a danger of falling into a body of water and 100% fall protection cannot be maintained. This includes barges, floats (without handrails), rowboats, motorboats, or any other equipment in or over water. **Life jackets will be worn at all times during electrofishing activities on Robinson Creek if water is greater than three feet deep and/or fast moving.**

When wearing a life jacket or work vest it should be adjusted and the top and bottom buckles fastened. Prior to and after each use, the life jacket or work vest must be inspected for defects which would alter their strength or buoyancy. Defective units must not be used.

### 5.2 ELECTROFISHING

#### 5.2.1 Introduction

In many cases, the most effective means of collecting fish for scientific purposes is electrofishing. Electrical current is placed in the water to immobilize fish, allowing them to be collected with dip-nets. It involves the use of either AC (alternating current) or DC (direct current) to immobilize fish for capture. These two types of current have very different effects on fish. The choice of current to use is dependent on the type of study being performed and the importance of returning healthy fish to the water. For the Marathon Robinson DELTs study, electrofishing will be used to conduct select elements of the field collections on Robinson Creek.

##### 5.2.1.1 AC & DC Current

AC current typically has the most violent effect on fish. Once in the electrical field a fish will immediately “take a posture transverse to the current in such a way as to receive a minimum of voltage” (Smith-Root). This action is called oscillotaxis. Fish will be immobilized quickly and the effect will last longer than that of DC current. Great care must be taken in the collection of fish in this manner. For the Robinson Creek field collections, we will be using DC electrofishing.

With DC current, fish react in three ways: first, they line up with the direction of the electrical current, then swim toward the anode (positive electrode). This reaction is called galvanotaxis. Finally, when fish near the anode they are stunned, roll belly up, and collection becomes possible. The effects of DC current do not last as long as AC current. When the power is turned off the fish recover quickly. Mortality is far more limited than with the use of AC. This,

along with the fact that fish actually swim to the anode, makes DC current the more effective means of electrofishing.

### **5.2.1.2 Control Box**

DC current can be selected with electrofishing control box. In addition to controlling the type of current, a control box allows adjustments to how the current acts. Most equipment will allow you to select for standard or pulsed output and to vary the pulse width and frequency of pulses, which allows for more efficient collections and limits the risk and stress to fish.

The control box also allows selection of voltage output. Depending on the electrofishing system used (i.e., Smith-Root), this selector should be positioned at the lowest possible setting that allows 5-10 amps to be obtained by adjusting the pulse width and rate or a minimum of 190 volts.

Pulsed output means that the electrical current going from the system into the water comes in pulses or waves. When the pulse rate is low and the width of the field is narrow, less current is required to collect fish. This results in less stress to fish. Since conductivity of water (the ease with which an electrical charge passes through it) varies, it is necessary to have the ability to adjust the pulse rate and width for optimum collection with minimum harm to the fish being collected.

### **5.2.1.3 Conductivity**

Electrofishing works by passing electrical current through a fishes body causing the effects described above. Several factors affect the amount of current passing through the fish's body and thus, the effectiveness of electrofishing. If the conductivity of the fish's body is equal to or slightly above the conductivity of the surrounding water, the electricity will choose the path of least resistance and pass through the fish. The greater the conductivity of the fish's body in relation to the surrounding water, the greater the effect of the electricity on the fish. The conductivity of fish flesh differs among species. When shocking, you may observe catfish floating up as far as 50 ft. from the anode. At the same time, scaled fish may not succumb to the current until they actually pass within a few feet of the anode. Also, due to increased surface area, larger fish, particularly large and deep-bodied fish, tend to receive a larger charge of electricity than do smaller fish.

Another factor that influences the effectiveness of electroshocking is the conductivity of the water. Pure distilled water will actually act as an insulator in an electrical current. This is because there are few electrolytes or dissolved solids to conduct the electricity. It would take a great deal of current to pass through this type of water. Conversely, the water of a typical lake or river may be very high in dissolved solids. This water will readily conduct very low amounts of current. In all cases, the conductivity of the water must be equal to or below the conductivity of the fishes body for electrofishing to be effective. It is not effective to shock in salt water because it is an electrolyte solution. The conductivity of the water is so much higher than that of a fish that an electrical current will find that the path of least resistance is actually around the fish rather than through it.

Conductivity of the water being surveyed should always be checked before attempting electrofishing. If it is very low ( $<50 \mu\text{S}/\text{cm}$ ) or extremely high ( $>4500 \mu\text{S}/\text{cm}$ ), a different type of collection should be considered. When backpack, pram (tow barge), or long line (bank unit) shocking small streams, it may actually be possible to increase the conductivity of the water by placing a block of salt upstream of the study area several hours before beginning your survey. This however, should only be considered in controlled conditions.

#### **5.2.1.4 Types of Equipment**

There are several types of electrofishing equipment available. EA typically uses boat, backpack, pram or long line units. These units differ in the type of power source used and in their application. For the Robinson Creek field collections, EA will use pram and/or long line wadeable units while wading in Robinson Creek.

Pram and long line electrofishing are designed for use in areas where boat electrofishing may not be possible or practical. Pram shocking involves the use of a power source and electroshocking unit placed in a barge or small boat. Like backpack electrofishing, the operator utilizes a hand held anode and trail behind cathode to place current in the water.

Long line electrofishing involves the use of a power source and electroshocking unit deployed on the bank. Like the other wadeable methods, the operator utilizes a hand held anode. However, the cathode is stationary, typically deployed in the middle of an electrofishing zone near the control box and power source, to place current in the water. As with the pram unit, the operator is not required to carry the power source and control box. Cables with up to 100m long allow mobility over a large section of water.

In all types of electrofishing, current is passed through the water between a positive electrode (anode) and a negative electrode (cathode). EA typically uses a boom mounted anode and the boat hull as a cathode when boat electrofishing. You may however, see different arrangements. In backpack electrofishing the anode is a hand-held probe or dipnet and the cathode is a trail behind cable. In pram shocking, the cathode may be the hull of either the barge or boat carrying the equipment, and in long line shocking the cathode is a cable or plate deployed from a bank-mounted power source.

#### **5.2.1.5 Equipment Operation**

Pram and long line shocking are slightly more hazardous than boat shocking because of the user's position in the water with the electrical charge. EA will utilize only experienced staff with several years of experience with the equipment and conducting the same type of work as will be done for the Robinson Creek field collections. For staff and visitors that are new, a field brief or field training sessions will be completed, as needed, before initiating the work.

Basically, a wadeable system is a miniaturized version of the boat electrofishing system. At least two operators are required for pram electrofishing while three operators are preferable when using the long line method. For pram shocking, the operator handles the anode, which consists of

a probe or a combination of probe and dipnet, depending on conditions. The second person monitors the equipment while assisting with the collection, transfer, and care of fish. For long line electrofishing, a third person typically maintains the cable and manages the live car.

The operator wades in an upstream direction through the water sweeping the anode 2-3 feet ahead. A thumb switch on the handle of the probe serves the same safety function as the foot switch on the boat. With a net probe, when a fish is shocked, the operator collects it with the dipnet, releases (i.e., turns off) the switch on the handle and places the fish in a bucket, live-well, or live car. If the anode is not operated with an attached net, the second person will closely follow the operator and anode with a dipnet to collect fish. When pram shocking, special attention should be paid by all crewmembers to the size of the electrical field. If the cathode is mounted on a barge, boat, or bank the electrical field will reach from that point to the anode held by the operator.

## **5.2.2 Safety**

### **5.2.2.1 Safety Awareness**

For the Robinson Creek field collections electrofishing will be performed by a trained field crews, with well-maintained equipment, electrofishing can be a very safe means to collect fish for biological study. Nonetheless, attention to safety must be paramount for all crew members in order to conduct a successful electrofishing survey. The amount of current in the water may be in excess of 250 volts. The amount of amperage generated during typical shocking operations averages 8 amps. This is enough to harm people, under certain circumstances, if the field were to come in direct skin contact with an electrical source such as a cathode, anode over a significant portion of the body (e.g., falling into and completely submerging in a strong electrical field at a close proximity to the sources mentioned or in concert with select medical conditions).

Therefore, awareness of the hazards, proper PPE, experience, and caution are paramount to the safe operation of electrofishing equipment.

### **5.2.2.2 Hazard Awareness**

Various physical hazards will potentially be present during electrofishing activities. These physical hazards may include, but may not be limited to:

- Working over, near, or in the water
- Slip, trip, and fall
- Weather
- Material handling, moving, lifting
- Fire/explosion
- Exposure (e.g., cold stress, heat stress, sun burn)
- Noise
- Electrical
- Biological (e.g., fish spine puncture wounds, poisonous insects and plants)

### 5.2.2.3 Safety Rules

Always follow the manufacturer's instructions when installing or operating electrical equipment. It is each crew leader's responsibility to familiarize crew members with the equipment and how to operate it. Furthermore, it is the responsibility of each crew member to assure that others are following proper procedures. **If EA staff are asked to do something that they feel is improper or unsafe, all have the authority to refuse and stop work.** Don't depend on someone else to look out for you. Look out for yourself.

Despite all of this, as mentioned above, electroshocking surveys can be conducted in a safe manner. All that is required is proper attention to detail and the use of the safety equipment provided.

The following are the primary common-sense rules that must be followed by all crew members at all times:

1. Life jackets are required to be worn at all times when water depth is greater than three feet and/or electrofishing is being conducted in fast moving water.
2. Prior to initiating a survey, the crew leader will conduct a safety briefing to remind or instruct support personnel on basic operation, safety, and hazard awareness. Prior to electrofishing at a given site, the crew will survey the study zone for potential hazards.
3. Wear rubber gloves when operating/touching electroshocking equipment.
4. Non-breathable, chest waders will be worn by all crew members for wadeable electrofishing
5. When conducting wadeable electrofishing, all equipment in the water (e.g, nets, live cars, live wells, buckets) must be non-conductive, insulated, and/or isolated.
6. Due to the conductive nature and added weight, steel-toed boots and/or weight belts must never be worn while electrofishing.
7. Lug-soled boots are appropriate when wading in soft and or fine substrates (e.g., silt, much, gravel, cobble). However, large and firm substrates (e.g., bedrock, boulder, large cobble) may be especially slippery and may require felt-soled wading boots or corkers to safely wade.
8. Never touch a loose wire or make an adjustment while unit is in operation. Rubber gloves must be worn, safety switches must be released, and the control box turned off before making any output adjustments. For all other system adjustments, beyond source, output, and other fine tuning at the control box, the power source and system must be shut-down, completely.
9. Always use safety switches. Never disable a safety switch or use equipment with an inoperable safety switch.
10. Never over-extend yourself when netting fish.
11. When wading, walk deliberately and carefully with a shuffling, wide stance to avoid unseen trip hazards.
12. Communicate hazards to fellow wading crew members. Each crew member has limits to their view. Don't assume everyone sees what you see. If noise level restricts normal conversation, establish hand signals.



13. Never place your bare hand in the water.
14. Look up from the water frequently to assure that overhanging branches or other items don't pose a risk.
15. If necessary, particularly for boat or pram operated equipment, wear hearing protection.
16. Maintain the equipment through routine maintenance checks. If repairs are needed, get them fixed immediately. Don't wait for the next person to do it.
17. Life jackets are not recommended for wadeable electrofishing in shallow water (i.e., less than three feet deep) as they restrict movement and may contribute to heat stress. In doing so, life jackets with shallow water, wadeable electrofishing actually present a greater hazard than wading without a life jacket.
18. Cold weather, dress warmly in layers of reasonably tight-fitting materials. Additional protections may include glove liners, hats, hand and foot warmers. For warm weather, light colored and light weight synthetic, quick dry fabrics should be worn. Maintain hydration and use sunscreen liberally to protect from sun burn.

Robinson Creek is a shallow stream with most working areas no deeper than two feet. In the event a staff member was to fall, in most cases, they can self-recover to their feet or be assisted by other electrofishing crew members. In the event a staff member becomes incapacitated, first, get the individual's face out of the water, check condition, and, if necessary, call 911 for emergency assistance. As needed, perform first aid and CPR.

### **5.3 WEATHER HAZARDS**

Weather conditions will be taken into consideration during each sampling effort. Heavy rains, electrical storms, high winds, and extreme temperatures, for example, may create extremely dangerous situations for employees. Inclement weather may also impair equipment performance. Whenever unfavorable conditions arise, the Crew Leader will evaluate both the safety hazards and ability of the employees to effectively perform given tasks under such conditions. Outdoor work will be suspended during thunderstorms, tornadoes, hurricanes, or other severe weather events. Weather conditions will be monitored via cell phone to identify the approach of severe weather situations.

### **5.4 HEAT STRESS**

Prolonged exposure to heat can result in heat rash (prickly heat), heat cramps, heat exhaustion, or heat stroke. Heat stroke is life threatening and requires immediate professional medical attention. An overview of these heat-induced illnesses and proper preventative actions are described below.

#### **5.4.1 Heat Rash (Prickly Heat)**

Heat rash, which is commonly observed in tropical climates, is a painful temporary condition caused by clogged sweat pores, typically from sleeping in hot, humid quarters. Heat rash appears as tiny red bumps on the skin and can impair sweating, resulting in diminished heat tolerance.

Heat rash can usually be cured by providing cool sleeping quarters; body powder may also help absorb moisture.

#### **5.4.2 Heat Cramps**

Heat cramps are characterized by painful intermittent spasms of the voluntary muscles following hard physical work in a hot environment. Heat cramps usually occur after heavy sweating and often begin towards the end of the workday. The cramps are caused by a loss of electrolytes, principally salt. This results in fluids leaving the blood and collecting in muscle tissue, resulting in painful spasms. Treatment consists of increased ingestion of commercially available electrolytic “sports” drinks (because of individual sensitivity, it is best to dilute by doubling the amount of water required by package directions or add water to the liquid form).

#### **5.4.3 Heat Exhaustion**

This condition is characterized by profuse sweating, weakness, low blood pressure, rapid pulse, dizziness, and frequently nausea and/or headache. The skin is cool and clammy and appears pale. The body core temperature is normal or depressed. Victim may faint and/or vomit. This is the most common work-related heat illness, and usually occurs after an extended period of work – look for signs of onset after lunch – an employee may suddenly need to sit down, feel faint, weak, or nauseated.

First aid consists of placing the victim in a cool area, loosening clothing, placing in a head-low (shock prevention) position, and providing rest and plenty of fluids. Any worker who is a victim of heat exhaustion may not be exposed to a hot working environment for an absolute minimum of 24 hours, and if fainting has occurred, the victim should not return to any work until authorized by a physician.

#### **5.4.4 Heat Stroke**

This is the most serious heat disorder, is life threatening, and is a true medical emergency. It results when the body’s heat dissipating system is overwhelmed and shuts down (thermoregulatory failure). Heat stroke results in a continual rise in the victim’s deep core body temperature, which is fatal if not checked. The symptoms are hot, dry, flushed skin, elevated body core temperature, convulsions, delirium, unconsciousness, and possibly death.

First aid consists of immediately moving victim to a cool area; cool the body rapidly by immersion in cool (not cold) water or sponging the body with cool water; treat for shock and obtain immediate medical assistance. Treatment response time is critical when assisting a victim of heat stroke! Do not give coffee, tea, or alcoholic beverages.

#### **5.4.5 Preventative Measures**

Unfortunately, there are no known PPE to prevent heat-related illnesses. However, some preventative measures to avoid heat stress include:

- Frequent resting in cool or shaded areas,
- Consumption of large quantities of potable water or diluted electrolyte beverages, following the suggested hydration target in Table 7:

**Table 7. Hydration Targets based on Air Temperature and Time Periods between Breaks**

Temperature	Work Level	Maximum Minutes Worked Between Hydration Breaks	Hydration Target
<80°F	Normal	--	8-12 oz./hr.
80-85°F	Normal	--	8-16 oz./hr.
86-90°F	Normal	50	12-20 oz./hr.
91-95°F	Normal	45	16-24 oz./hr.
>96°F	Normal	40	24-32 oz./hr.

- Following a work/rest regiment from Table 8:

**Table 8. Work/Rest Schedule based on Air Temperature**

Ambient Temperature	Work (hours)	Rest (minutes)
70°F	3	15
75°F	2½	15
80°F	2	15
85°F	1½	15
90°F	1	15

Other factors, such as a worker's acclimatization, level of physical fitness, and age, may increase or decrease his/her susceptibility to heat stress. Before assigning a task to an individual worker, these factors will be considered to ensure that the task will not endanger the worker's health.

If a heat-related illness is suspected or observed, the affected person must be moved to a cool or shaded area and given plenty of liquids to consume. If symptoms of a heat stroke are observed, the victim will be cooled immediately and treated as a medical emergency. Liquids will be readily available to ensure that workers stay hydrated.

## 5.5 BIOLOGICAL HAZARDS – INSECT BITES/STINGS

Protective outer clothing such as gloves, hard hats, and coveralls can reduce the potential for insect bites and stings. Insect bite symptoms may include redness, rash, swelling, chills, fever, diarrhea, and vomiting. Any worker who has been bitten or stung and shows symptoms of a severe reaction should seek medical assistance immediately. Workers who know of their allergies to insects should advise their supervisor prior to field activities and should carry an antidote kit, if necessary.

When working in areas near heavy vegetation (possibly the riverbank) and to prevent contact with disease-carrying ticks, workers should wear long-sleeved shirts, long pants, and boots that extend above the ankle with socks pulled over pant cuffs or with pants legs taped to boots. Insect repellent is also an effective means of tick control. Workers should check clothing, skin,

and hair for the presence of ticks periodically and thoroughly at the end of each workday. If a tick attaches to the body, it should be removed by gently tugging with tweezers where the mouth enters the skin. The tick should not be killed prior to removal.

## **5.6 BIOLOGICAL HAZARDS – FISH**

Care must be taken and the proper PPE used when handling certain fish species to avoid getting cut, spined, or bitten. Proper handling of fish will be performed by experienced project personnel to reduce the likelihood of injury. Any worker that gets injured from a fish should seek medical attention immediately.

## **5.7 POISON IVY AND RELATED PLANTS**

Poison ivy, poison oak, and poison sumac have poisonous sap (urushiol) in their roots, stems, leaves, and fruits. The urushiol may be deposited on the skin by direct contact with the plant or by contact with contaminated objects, such as clothing, shoes, tools, and animals. Preventative measures include: wear long-sleeved shirts and long pants tucked into boots; wear cloth or leather gloves; apply barrier creams (e.g., Ivy Block) to exposed skin; and be able to identify poison ivy, oak, and sumac plants. If you are exposed, according to the U.S. Food and Drug Administration (FDA), you should quickly (within 10 minutes): 1) cleanse exposed areas with rubbing alcohol; 2) wash the exposed areas with water only (no soap yet, since soap can move the urushiol, which is the oil from the poison ivy that triggers the rash, around your body and actually make the reaction worse); 3) take a shower with soap and warm water; and 4) put gloves on and wipe everything you had with you, including shoes, tools, and your clothes, with rubbing alcohol and water.

Unfortunately, if you wait more than 10 minutes, the urushiol will likely stay on your skin and trigger the poison ivy rash. You may not be able to stop it on your skin, but you might still scrub your nails and wipe off your shoes, etc., so that you do not spread the urushiol to new areas.

## **5.8 ALLERGIC REACTIONS**

When in the field, personnel may be exposed to allergens that can cause mild to severe allergic reactions. The following guidelines will explain how to help a person having an allergic reaction.

For a mild to moderate reaction:

- Calm and reassure the person having the reaction, as anxiety can worsen symptoms.
- Try to identify the allergen and have the person avoid further contact with it. If the allergic reaction is from a bee sting, scrape the stinger off the skin with something firm (such as a fingernail or plastic credit card). Do not use tweezers; squeezing the stinger will release more venom.
- If the person develops an itchy rash, apply calamine lotion and cool compresses.

Avoid medicated lotions.

- Watch the person for signs of increasing distress.
- Get medical help. For a mild reaction, a physician may recommend over-the-counter medications (such as antihistamines).

For a severe allergic reaction:

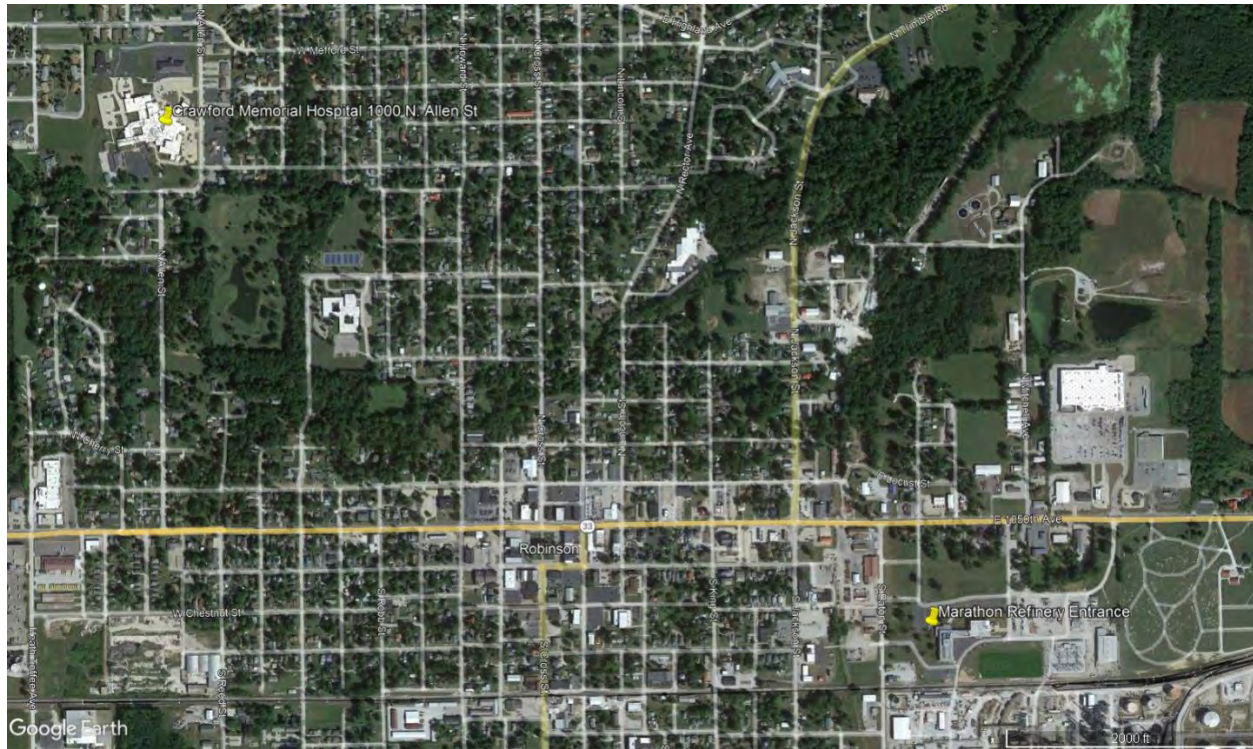
- Check the person's airway, breathing, and circulation (the ABCs of Basic Life Support). A warning sign for dangerous throat swelling is a very hoarse, whispered voice or coarse sounds when the person is breathing air in. If the victim is having difficulty breathing, is very weak, or is losing consciousness, call for emergency medical assistance.
- Calm and reassure the person.
- If the person has emergency allergy medication on hand, help the person take or inject the medication. Avoid oral medication if the person is having difficulty breathing.
- Take steps to prevent shock. Have the person lie flat, elevate the person's feet about 12- inches, and cover him or her with a coat or blanket. **DO NOT** place the person in this position if a head, neck, back, or leg injury is suspected or if it causes discomfort.

## **5.9 EMERGENCY RESPONSE PLAN**

In the event of an emergency, the information available at the time must be properly evaluated and the appropriate steps taken to implement the emergency response plan. The Crew Leader or senior onsite supervisor will assume command of the situation and call 618-544-2121 Ext. 5300 inside the refinery or 911 outside the refinery from the nearest telephone or cell phone, to notify authorities of your location (the docking point at which you will meet them), evacuate personnel as needed, and take other steps needed to gain control of the emergency.

Appropriate first aid will be given and emergency contacts will be made. Emergency situations will be handled by offsite support personnel; however, initial response and first aid will be available from qualified onsite personnel. Once the situation is under control, the Crew Leader or designee will immediately call EA's Corporate Safety and Health Officer (Rob Marcuse at 410-329-5192) and must complete an Accident/Loss Report.

The nearest hospital to the project site is Crawford Memorial Hospital, located at 1000 N. Allen Street, Robinson, Illinois 62454 (618-544-3131).



**Figure 6. Location of Crawford Memorial Hospital and MPC Entrance.**

### **5.10 CORONAVIRUS DISEASE 2019 (COVID-19)**

All EA staff will be fully vaccinated against COVID-19 prior to arrival on-site. EA staff will adhere to current state and/or local PPE and social distancing requirements, as necessary. Appropriate PPE, including face masks and hand sanitizer, will be available to EA staff as needed.

**6. COMMUNICATION AND CONTACT INFORMATION**

<b>LOCAL EMERGENCY TELEPHONE NUMBERS</b>	
Crawford Co. Sherriff	911 or (618) 546-1515
Robinson Police Department (Robinson, IL)	911 or (618) 544-2217
Robinson Township Fire Dept. (Robinson, IL)	911 or (618) 544-2955
Crawford Memorial Hospital (Robinson, IL)	(618)-544-3131
Poison Control Center	(800) 492-2414 or (800) 222-1222
<b>Region 5 Department of Natural Resources Personnel (618) 435-8138</b>	
Boone LaHood - Fisheries Biologist	Office (O): (618) 393-6732
Logan Willand - Area Sgt. (Law Enforcement)	(779) 970-0234
<b>PROJECT-RELATED TELEPHONE NUMBERS</b>	
EA Engineering, Science, and Technology, Inc., PBC (HV)	(410) 584-7000
Joe Vondruska, STR (EA)	O: (847) 607-6485/C: (847) 271-8412
Rob Marcase, Health and Safety (EA)	O: (410) 329-5192/C: (717) 586-9878
Michele Bailey, Human Resources (EA)	O: (410) 527-2481/C: (410) 790-3795
Jeff Boltz, WNR Director (EA)	O: (410) 329-5179/C: (410) 804-9230
Marty Sneen, Scientist, Project Manager (EA)	O: (847) 607-6484/C: (847) 372-6332
Ken Cummings, Scientist, Field Lead (EA)	O: (847) 607-6475/C: (847) 271-8406
Marisol Sepúlveda, Scientist, Fish Health (Purdue)	(765) 496-3428
Michael Chanov, Scientist, Bioassay Lead (EA)	410 329-5120
Julie Holscher, MPC Environmental	(618) 469-5336
Lisa Stewart, MPC – Safety Training	(618) 546-5111
Lenzi Ippolito, MPC Environmental	O: (618)-469-5553/C: (618) 553-0144
Lisa Stewart, MPC Orientation	618-546-5111
MPC Emergency	(618)-544-2121 ext. 5300
Contract Laboratory	Ken Hunt (317) 228-3120 at Pace Indianapolis (Kenneth.Hunt@pacelabs.com)
UPS	1-800-742-5877
Federal Express	6.1.1.1 (1-800-GO-FEDEX)

## 7. REFERENCES

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Slaninova A., M. Smutna, H. Modra, and Z. Svobodova. 2009. A review: Oxidative stress in fish induced by pesticides. *Neuroendocrinology Letters* 30: Suppl. 1.



Starke A., A. Haudum, R. Busche, M. Beyerbach, S. Dänicke, and J. Rehage. 2010. Analysis of total lipid and triacylglycerol content in small liver biopsy samples in cattle. *Journal of Animal Sciences* 88:2741–2750.

United States Environmental Protection Agency. 2004. Quality Assurance/Quality Control Guidance for Laboratories Performing PCR Analyses on Environmental Samples. Office of Water (4607) EPA 815-B-04-001.

Van der Laan, R., R. Fricke, and W.N. Eschmeyer (eds). 2022. ESCHMEYER'S CATALOG OF FISHES: CLASSIFICATION. (<http://www.calacademy.org/scientists/catalog-of-fishes-classification/>). Electronic version accessed 14 June 2022.

**Appendix A**  
**Standard Operating Procedure for Surface Water**  
**Sampling**

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**Appendix A**  
**Standard Operating Procedure for Surface Water**  
**Sampling**

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**Standard Operating Procedure No. 007  
for  
Surface Water Sampling**

*Prepared by*

EA Engineering, Science, and Technology, Inc., PBC  
225 Schilling Circle, Suite 400  
Hunt Valley, Maryland 21031

Revision: 02  
March 2020

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**PROJECT-SPECIFIC VARIANCE FORM**

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

**A. Variances required; cite section(s) of the SOP to which there is a variance**

**B. No variances**

SOP No. 007	
SOP Section	Variance

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Project Manager (Name)

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Project Manager (Signature)

Date



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**DOCUMENT REVISION HISTORY**

<b>ORIGINAL (MASTER) DOCUMENT REVISION HISTORY</b>				
<b>Revision Number</b>	<b>Revision Date</b>	<b>Revision Summary</b>	<b>Revised By</b>	<b>Reviewed By</b>
02	March 2020	Systematic Review and Update	Jason Stroup Kevin Kowalk	Matthew Bowman

## 1. SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to delineate protocols for sampling surface water. This procedure can be applied to the collection of surface water samples from marine and estuarine systems, streams, rivers, ditches, lakes, ponds, and lagoons. Surface water samples provide an indication of the amount of contaminant in the surface water. It is, therefore, important to collect a representative sample.

## 2. MATERIALS

The following materials may be required:

0.45-micrometer ( $\mu\text{m}$ ) disposable filters	Sample bottles
Cooler with ice	Short-handled dip sampler (PTFE or stainless steel)
Long-handled dip sampler (polytetrafluoroethylene [PTFE] or stainless steel)	Stainless steel or PTFE-lined bucket
Peristaltic pump	Niskin bottle (or similar sampling device)
Disposable peristaltic head tubing	Disposable Teflon and/or Teflon lined poly tubing

## 3. PROCEDURE

For all surface water samples, use a Global Positioning System to record sampling coordinates and mark the sampling locations on a site map. Photograph (if cameras are allowed onsite) and describe each location, place a numbered stake above the visible high-water mark on the bank closest to the sampling location, and/or mark adjacent trees with surveyor's flagging. The photographs and descriptions must be adequate to allow the sampling station to be relocated at some future date by someone other than the original sampling crew. Use a long-handled dip sampler where access is poor or non-contact with water is suggested in the Health and Safety Plan.

Sampling should be performed deliberately and methodically to minimize disturbance of bottom sediments, yet as quickly as possible to ensure a representative sample. If wading in a stream, sample upstream at the sampling location to prevent disturbance of the stream bottom from impacting the sample. To prevent contamination of the exterior of the sample container, and/or potential contamination of the surface water sample by laboratory contaminants on the exterior of the bottle, the sample container should never be dipped into the water; rather, a decontaminated sampling device should be used to collect unfiltered samples.

Sampling with the PTFE or stainless steel sampler (long-handled or measuring cup-type):

- Remove the cap from the sample bottle.
- Dip a sample of surface water using the sampler.

- Tilt sample bottle and gently pour sample from sampler into the bottle. Allow the sample to trickle down the side of the bottle. Avoid aerating the sample.
- Add preservative as required. Replace cap, and place in cooler immediately.

Sampling with stainless steel or PTFE-lined bucket:

- Remove cap from sample bottle.
- Gently dip collection bucket in the water. Fill bucket and carefully lift from water body.
- Tilt sample bottle and gently pour sample from sampler into the bottle. Allow the sample to trickle down the side of the bottle. Avoid aerating the sample.
- Add preservative as required. Replace cap, and place in cooler immediately.

– **OR** –

- Use smaller sampling cup to transfer sample from bucket to sample bottle as described above.

Sampling with a Niskin bottle (or similar device):

- Prepare the bottle for deployment by placing the ends of the bottle in the open position and lock the ends into the trigger mechanism.
- Lower the bottle to the desired depth of sampling (on either a wire cable or rope).
- Place a messenger (triggering device) on the cable/rope and deploy by allowing free-fall down the cable/rope.
- Bring the bottle back to the surface and pour sample into a sample container.

Sampling with a peristaltic pump and Teflon<sup>®</sup> or Teflon<sup>®</sup> lined tubing:

- Cut a length of Teflon<sup>®</sup> tubing to the depth of sampling specified by the client or project-specific Sampling and Analysis Plan.
- Insert one end of the tubing into the intake hose on the peristaltic pump.
- Place a stainless steel weight on the tubing and lower to the specified depth;
- Cut a length of tubing and insert into the output (out-flow) hose on the peristaltic pump.

- After applying power to the peristaltic pump, proceed to pump site water through the tubing apparatus. The hose volume should be pumped approximately five times through the tubing before sampling.
- Fill the required sample containers.
- If filtering is required, obtain filtered sample by placing a 0.45- $\mu\text{m}$  in-line filter on the end of the output tube and fill the required sample containers.

Both filtered and unfiltered samples may be required for metals analyses. Bulk samples for filtration will be collected using the stainless steel or PTFE-lined bucket method described above. Sample filtration must be performed immediately upon retrieval of the bulk sample as follows.

Filtration will be performed immediately after collecting sample. Set up filtration equipment prior to collecting sample. Filtration may be accomplished by gravity or, if necessary due to slow filtering, a peristaltic pump will be used to pressure filter the sample. Vacuum filtration will not be used due to the possibility of analyte volatilization.

Gravity filtration will be accomplished as follows:

- Using decontaminated forceps, place a 0.45- $\mu\text{m}$  membrane in a decontaminated filter funnel.
- Slowly pour sample into the funnel and collect filtrate directly into appropriate sample container(s).
- Add preservative(s) as required by project-specific Sampling and Analysis Plan. Immediately cap container and place in cooler.
- Dispose of filter membrane.

Pressure filtration will be accomplished as follows:

- Using previously assembled disposable tubing, 45- $\mu\text{m}$  in-line filter, and peristaltic pump, filter sample from collection bucket into appropriate container.
- Adjust pump rate to avoid aeration of sample.
- Fill container, add preservative as required immediately cap container, and place in cooler.
- Dispose of filter and tubing.

#### **4. MAINTENANCE**

Refer to manufacturer's specifications for maintenance procedures on generators and pumps.

#### **5. PRECAUTIONS**

The following precautions should be taken:

- Avoid disturbing bottom sediments.
- Consult the Health and Safety Plan prior to collecting any samples for personal protective equipment such as dermal and respiratory protection and personal flotation devices when sampling in or near deep water or from boats.
- Always decontaminate the sampling and filtration equipment, and change gloves between sampling locations to minimize the risk of cross-contamination.

#### **6. REFERENCES**

None.

**Appendix B**  
**Standard Operating Procedure for Sample  
Preservation and Container Requirements**



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**Standard Operating Procedure No. 039**  
**for**  
**Sample Preservation and**  
**Container Requirements**

*Prepared by*

EA Engineering, Science, and Technology, Inc., PBC  
225 Schilling Circle, Suite 400  
Hunt Valley, Maryland 21031

Revision 2  
September 2018

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**PROJECT-SPECIFIC VARIANCE FORM**

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

- A. Variances required; cite section(s) of the SOP to which there is a variance**
- B. No variances**

SOP No. 039	
SOP Section	Variance

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Project Manager (Name)

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Project Manager (Signature) Date

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**DOCUMENT REVISION HISTORY**

<b>ORIGINAL (MASTER) DOCUMENT REVISION HISTORY</b>				
<b>Revision Number</b>	<b>Revision Date</b>	<b>Revision Summary</b>	<b>Revised By</b>	<b>Reviewed By</b>
2	25 September 2018	Add notes about incremental sampling and minor changes	Daniel Hinckley, Sanita Corum	Matthew Bowman



## 1. PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure (SOP) is to define the preservatives and techniques to be employed in preserving environmental samples between collection and analysis.

## 2. MATERIALS

The following materials may be required:

- Containers (Section 3 provides a description)
- Nitric acid
- Sulfuric acid
- Sodium hydroxide
- Ice chests
- Ice.

## 3. DEFINITION OF CONTAINER TYPES

Listed below are the definitions of various container types.

Type	Container	Closure	Septum
<b>A</b>	80-ounce amber glass, ring handle bottle/jug, 38-millimeter (mm) neck finish	White polypropylene or black phenolic, baked polyethylene cap, 38-430 size, 0.015-mm polytetrafluoroethylene (PTFE) liner	
<b>B</b>	40-milliliter glass vial, 24-mm neck finish	White polypropylene or black phenolic, open top, screw cap, 15-mm opening, 24-400 size	24-mm disc of 0.005-inch) PTFE bonded to 0.120-inch silicon for total thickness of 0.125 inches
<b>C</b>	1-liter high density polyethylene, cylinder-round bottle, 28-mm neck finish	White polyethylene cap, white ribbed, 28-410 size; F217 polyethylene liner	
<b>D</b>	120-milliliter wide mouth glass vial, 48-mm neck finish	White polyethylene cap, 40-480 size; 0.015-mm PTFE liner	
<b>E</b>	250-milliliter Boston round glass bottle	White polypropylene or black phenolic, open top, screw cap	Disc of 0.005-inch PTFE bonded to 0.120-inch silicon for total thickness of 0.125 inches
<b>F</b>	8-ounce short, wide mouth, straight-sided, flint glass jar, 70-mm neck finish	White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.030-mm PTFE liner	
<b>G</b>	4-ounce tall, wide mouth, straight-sided, flint glass jar, 48-mm neck finish	White polypropylene or black phenolic, baked polyethylene cap, 48-400 size; 0.015-mm PTFE liner	



Type	Container	Closure	Septum
<b>H</b>	1-liter amber, Boston round, glass bottle, 33-mm pour-out neck finish	White polypropylene or black phenolic, baked polyethylene cap, 33-430 size; 0.015-mm PTFE liner	
<b>K</b>	4-liter amber glass ring handle bottle/jug, 38-mm neck finish.	White polypropylene or black phenolic, baked polyethylene cap, 38-430 size; 0.015-mm PTFE liner	
<b>L</b>	500-milliliter high-density polyethylene, cylinder bottle, 28-mm neck finish	White polypropylene, white ribbed, 28-410 size; F217 polyethylene liner	

#### 4. PROCEDURE

All containers described in Section 3 must be certified clean (SOP Number [No.] 031), with copies of laboratory certification furnished upon request. There may be circumstances when alternative containers will be used (e.g., aluminum foil around tissue samples placed in plastic bags, plastic buckets or bags for large soil/sediment samples, etc.) for which laboratory certification may not be available. Such containering should be appropriately decontaminated or verified appropriately clean prior to using.

Water samples will be collected into pre-preserved containers appropriate to the intended analyte as documented in the Quality Assurance Project Plan. Samples taken for volatile organic compounds will be collected in accordance with SOP No. 003, Section 3.3.8. Samples taken for metals analysis will be verified in the field to a pH <2. The container should be tightly capped, then swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure that this operation may have generated. Samples taken for total phosphorous content will be verified in the field to a pH <2. The container should be tightly capped and swirled to thoroughly mix the sample. The cap will then be loosened to release any excess pressure that this operation may have generated. Samples taken for cyanide will be verified for a pH >12. Most other samples do not require added preservation; however, there are analytes that may require special preservation, (i.e., sulfide that requires a zinc acetate preservation). Preservation must be performed as documented in the project-specific Quality Assurance Project Plan. These samples will be immediately placed on ice and cooled to 4±2 degrees Celsius (°C).

Soil and sediment samples will be collected into containers appropriate to the intended analyte as documented in the Quality Assurance Project Plan. Samples taken for volatile organic compound analysis will be collected in accordance with the site-specific SOP. Samples taken for metals analysis will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Samples taken for total phosphorous content will be tightly capped, placed on ice, and maintained at a temperature of 4°C. Large (1-2 kilograms) soil/sediment samples taken for incremental samples (SOP No. 057) can be placed in pre-cleaned (SOP No. 005) gallon plastic bags or plastic buckets. Under most circumstances, no preservatives will be added to soil or sediment samples; follow project-specific requirements as documented in the Quality Assurance Project Plan. These samples will be immediately placed on ice and cooled to 4±2°C.

## 5. MAINTENANCE

Not applicable.

## 6. PRECAUTIONS

Note that acidifying a sample containing cyanide may liberate hydrogen cyanide gas.

- Avoid breathing any fumes emanating from acidified samples.
- Acidify samples only in the open, rather than in closed spaces (i.e., a vehicle).
- Hold suspected hydrogen cyanide-generating sample away from body and downwind while manipulating it.
- See the Health and Safety Plan for other safety measures.

## 7. REFERENCES

U.S. Environmental Protection Agency (EPA). 1986. Test Methods for Evaluating Solid Waste, SW-846.

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**Appendix C**  
**Standard Operating Procedure for**  
**Chain-of-Custody Form**

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**Standard Operating Procedure No. 002  
for  
Chain-of-Custody Form**

Prepared by

EA Engineering, Science, and Technology, Inc., PBC  
225 Schilling Circle, Suite 400  
Hunt Valley, Maryland 21031

Revision: 01  
November 2018

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**PROJECT-SPECIFIC VARIANCE FORM**

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

**A. Variances required; cite section(s) of the SOP to which there is a variance**

**B. No variances**

<b>SOP No. 002</b>	
<b>SOP Section</b>	<b>Variance</b>

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Project Manager (Name)

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Project Manager (Signature)

Date



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**DOCUMENT REVISION HISTORY**

<b>ORIGINAL (MASTER) DOCUMENT REVISION HISTORY</b>				
<b>Revision Number</b>	<b>Revision Date</b>	<b>Revision Summary</b>	<b>Revised By</b>	<b>Reviewed By</b>
01	November 2018	Systematic review and update	Dan Hinckley, Sheena Styger, Sanita Corum	Matthew Bowman



## 1. SCOPE AND APPLICATION

A chain-of-custody record (attached) is used as physical evidence of sample custody and as a permanent record for each sample collected. A chain-of-custody record documents the exchange and transportation of samples from the field to the laboratory. The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for use of the chain-of-custody form. Three example forms are provided as Figures SOP002-1 (EA's standard electronic chain-of-custody form), SOP002-2 (EA's Toxicology Laboratory chain-of-custody form), and SOP002-3 (U.S. Environmental Protection Agency [EPA] Scribe chain-of-custody form). Other formats with similar levels of detail are acceptable.

Most EPA projects utilize sampling and chain-of-custody instructions as documented in EPA's Samplers Guide (2014), which includes the use of Scribe, an in-house software program used to establish computer records of all environmental data and includes generation of chain-of-custodies. Using Scribe requires training, and the software and guidance can be found at the following link: [https://response.epa.gov/site/site\\_profile.aspx?site\\_id=ScribeGIS](https://response.epa.gov/site/site_profile.aspx?site_id=ScribeGIS). Training on Scribe is necessary and can be obtained through the Scribe weblink.

All new U.S. Army Corps of Engineers projects require the use of Formerly Used Defense Sites chemistry database (FUDSchem), which can be found at the following link: [http://fudschem.com/public/framework/bannerhtml.aspx?dsn=system&idhtml=10642&themesuffix=default&banner=banner\\_fudschem.jpg](http://fudschem.com/public/framework/bannerhtml.aspx?dsn=system&idhtml=10642&themesuffix=default&banner=banner_fudschem.jpg). This software will generate chain-of-custody forms specific to the sampling session. As with Scribe, FUDSchem training is necessary.

It is essential that chain-of-custody forms be completed properly, and that sample relinquishment be signed and dated appropriately. Laboratories use chain-of-custodies as their statement of work and, if it is not correct, the samples will not be analyzed appropriately. Sample custody documentation assures that the particular samples have been in secure locations, and that none of them have been tampered with, thus assuring appropriate results.

## 2. MATERIALS

The following materials may be required: chain-of-custody form and indelible ink pen.

## 3. PROCEDURE

- Give the site name and project name/number.
- Enter the sample identification code.
- Indicate the sampling dates for all samples.
- List the sampling times (military format) for all samples.

- Enter the total number of containers per cooler.
- List the analyses/container volume.
- Obtain the signature of sample team leader.
- State the carrier service and airbill number, analytical laboratory, and custody seal numbers (if applicable).
- Sign, date, and time the “relinquished by” section. Be sure the carrier signs and enters dates and time of acceptance of the samples.
- Upon completion of the form, retain a copy or portable document format, and affix the laboratory copy to the inside of the sample cooler in a zip-seal bag to protect from moisture, to be sent to the designated laboratory.

#### **4. MAINTENANCE**

Not applicable.

#### **5. PRECAUTIONS**

None.


#### **6. REFERENCES**

U.S. Environmental Protection Agency (EPA). 2014. Sampler’s Guide, Contract Laboratory Program Guidance for Field Samplers. EPA/540/R014/013, Directive 92400.2-147. October.

## Figures

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**Figure SOP002-1 EA Chain-of-Custody Form**

Company Name:				Project Manager or Contact:				Parameters/Method Numbers for Analysis												Chain-of-Custody Record		
Project No.				Phone:				No. of Containers													 EA Laboratories 231 Schilling Circle Hunt Valley, MD 21031 Telephone: (410) 584-7000	
Dept.:          Task:				Project Name:																		
Sample Storage Location:				P.O. No.:																		
Page of				Report No.:																	<b>Report Deliverables:</b> 1   2   3   4   D   E  EDD: Yes/No  DUE TO CLIENT: _____	
Date	Time	Water	Soil	Sample Identification 19 Characters				No. of Containers													EA Labs Accession Number	Remarks
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EA Engineering, Science, and Technology, Inc., PBC


Company Name:		Project Manager or Contact:		Parameters/Method Numbers for Analysis										 EA Laboratories 231 Schilling Circle Hunt Valley, MD 21031 Telephone: (410) 584-7000									
Project No.:		Phone:																					
Dept.:      Task:		Project Name:																					
Sample Storage Location:		P.O. No.:																					
Page    of		Report No.:																					
Date	Time	Water	Soil	Sample Identification 19 Characters	No. of Containers																EA Labs Accession Number	Remarks	
				XXXXXXXXXXXXXXXXXXXX																			
				XXXXXXXXXXXXXXXXXXXX																			
				XXXXXXXXXXXXXXXXXXXX																			
				XXXXXXXXXXXXXXXXXXXX																			
				XXXXXXXXXXXXXXXXXXXX																			
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				XXXXXXXXXXXXXXXXXXXX																			
Samples by: (Signature)			Date/Time	Relinquished by: (Signature)			Date/Time	Received by: (Signature)		Date/Time													
Relinquished by: (Signature)			Date/Time	Received by Laboratory: (Signature)			Date/Time	Airbill Number:		Sample Shipped by: (Circle)													
Cooler Temp.    C    pH:    Yes    No			Comments:			Custody Seals Intact    Yes    No			Fed Ex.    Puro.														
NOTE: Please indicate method number for analyses requested. This will help clarify any questions with laboratory techniques.										UPS													
										Hand Carried													
										Other:													



Figure SOP002-2 EA Toxicology Laboratory Chain-of-Custody Form

Client:		Project Manager:																	
		Phone:																	
		Project Contact:																	
		Phone:																	
Project Name:																			
Project#:																			
Page 1 of 1																			
Sample Collected		Matrix		No. of Containers															
SAMPLE IDENTIFICATION																			
Date	Time	Sediment	Water																
Sampled by: (Signature)		Date/Time		Relinquished by: (Signature)												Date/Time			
Relinquished by: (Signature)		Date/Time		Received by Laboratory: (Signature)								Date/Time							

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Figure SOP002-3 EPA Scribe Chain-of-Custody Form

Page 1 of 1

USEPA

Lab Contact: John Smith  
CarrierName: UPS  
AirbillNo:  
1Z2886820195095104

CHAIN OF CUSTODY RECORD

Site #: 47909  
Contact Name: Michael Smith  
Contact Phone: 800-332-0534

No: 5-112818-101859-0021

Cooler #: 13  
Case #: 47909  
Lab Phone: 800-660-1990

Lab #	Sample #	Location	Analyses	Matrix	Sample Date	Sample Time	Numb Cont	Container	Preservative	Lab QC
	HT18-01	HT18-01	MI 10 Metals + FE, NI 34PAHs, PCBs, TOC, Moisture	Sediment			1	8 oz amber	4 C	
	HT18-02	HT18-02	MI 10 Metals + FE, NI 34PAHs, PCBs, TOC, Moisture	Sediment			1	8 oz amber	4 C	
	HT18-03	HT18-03	MI 10 Metals + FE, NI 34PAHs, PCBs, TOC, Moisture	Sediment			1	8 oz amber	4 C	

Special Instructions:	SAMPLES TRANSFERRED FROM
	CHAIN OF CUSTODY #

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt



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**Appendix D**  
**Illinois Scientific Collection Permits**

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ILLINOIS DEPARTMENT OF NATURAL RESOURCES

Authorization is hereby granted, under 520 ILCS 5/3.22 and 515 ILCS 5/20-100 of the Illinois Compiled Statutes and 17 ILL. Adm. Code 520 to:

Last Name: **Bushing**

First Name: **Larry**

Permit Number: **A22.5273**

Issued: **3/4/2022**

Expires: **12/31/2022**

Business Name/Institution: EA Engineering, Science, and Technology, I

Street Address: 444 Lake Cook Rd, Suite 18

City: Deerfield, IL

Zip Code: 60015

for strictly scientific, educational or zoological purposes, to take the Illinois fauna identified below subject to the following provisions:

May legally capture, by scientifically accepted methods, only the specific aquatic life species listed on the accompanying Illinois Department of Natural Resources (IDNR) scientific permit application/project proposal (on file in Springfield, IL) strictly for scientific, educational, and/or zoological purposes (except endangered and threatened species). After data has been humanely collected from these species, all animals shall be released unharmed at or near the original site of capture. Deceased animals and/or animal parts must be buried or given to a public or state scientific educational or zoological institution. A federal permit is required for all projects involving federally regulated species. If endangered and threatened species are to be taken, the IDNR Division of Natural Heritage, Endangered Species Coordinator must be notified and must approve in writing all project related activities of the permit application.

Possession/Transportation of injurious aquatic life species requires appropriate permits in addition to the previously stated. The IDNR Aquaculture Specialist can be contacted to request and seek approval of all project activities of the permit applicant prior to activities being initiated. If such species are encountered as part of previously authorized projects, they may be kept for deposition into state, scientific, educational, or zoological institutions, if appropriate precautions are taken to further restrict potential release into the environment AND immediate reporting of escape to [dnr.aquaculture@illinois.gov](mailto:dnr.aquaculture@illinois.gov). (All aquatic life may be immediately returned unharmed from where they were taken. 515 ILCS 5/10-100.)


Authorization: Statewide, exclusive of nature preserves, and IDNR owned and managed properties

Individuals authorized to work under direct supervision of permittee: None


I agree to the following provisions and terms of this Scientific Permit.

Permittee's

Signature:

  
(Permit not valid unless signed)

Approved By:

  
Office of Resource Conservation

Date: 3/4/2022

**TERMS FOR SCIENTIFIC PERMIT**

1. This permit is valid only for the approved methods, locations and activities stated on the permit.
2. All permitted activities shall be performed by or under the direct supervision of the permittee. Permittee must be present with persons involved in actual taking of fauna.
3. Under no circumstances shall a scientific permit be used in lieu of sport or commercial licenses.
4. This permit is valid only for species not listed as Illinois Threatened or Endangered (<https://www.dnr.illinois.gov/conservation/NaturalHeritage/Pages/EndangeredandThreatenedSpecies.aspx>). If a Threatened or Endangered species is incidentally captured, the specimen must be released and the occurrence must be reported to [tara.kieninger@illinois.gov](mailto:tara.kieninger@illinois.gov) within 5 business days.
5. This permit does not allow the privilege of trespass. Landowner permission is required. Activities on Department sites are not permitted without the prior approval of the Site Superintendent. Activities on Illinois Nature Preserves and Land and Water Reserves must have prior approval from the Illinois Nature Preserve Commission.
6. Permittee must carry this permit at all times when taking specimens and be presented, upon request, to Department personnel.
7. Fauna taken and/or salvaged and rehabilitated must be released to the wild or permanently donated to a public or state scientific educational or zoological institution
8. This permit does not supersede Federal permits, which may be necessary for the permitted work.
9. All gear left unattended must be tagged bearing name and scientific permit number of permittee.
10. Use of rotenone or any other toxic materials for taking of fauna must have written approval from the Department prior to using such materials, and may need a variance from the Illinois Environmental Protection Agency.
11. An annual report must be submitted to the Department by January 31 of each year.
12. This permit may be revoked or suspended if the Department finds that a permittee has falsified information on the application, failed to comply with the provisions of this permit, or violated state or federal laws.

The Department of Natural Resources is an equal opportunity employer.



## ILLINOIS DEPARTMENT OF NATURAL RESOURCES

Authorization is hereby granted, under 520 ILCS 5/3.22 and 515 ILCS 5/20-100 of the Illinois Compiled Statutes and 17 ILL. Adm. Code 520 to:

Last Name: **Cummings**

First Name: **Ken**

Permit Number: **A22.0335**

Issued: **3/4/2022**

Expires: **12/31/2022**

Business Name/Institution: **EA Engineering, Science, and Technology, I**

Street Address: **444 Lake Cook Rd. Suite 18**

City: **Deerfield, IL**

Zip Code: **60015**

for strictly scientific, educational or zoological purposes, to take the Illinois fauna identified below subject to the following provisions:

May legally capture, by scientifically accepted methods, only the specific aquatic life species listed on the accompanying Illinois Department of Natural Resources (IDNR) scientific permit application/project proposal (on file in Springfield, IL) strictly for scientific, educational, and/or zoological purposes (except endangered and threatened species). After data has been humanely collected from these species, all animals shall be released unharmed at or near the original site of capture. Deceased animals and/or animal parts must be buried or given to a public or state scientific educational or zoological institution. A federal permit is required for all projects involving federally regulated species. If endangered and threatened species are to be taken, the IDNR Division of Natural Heritage, Endangered Species Coordinator must be notified and must approve in writing all project related activities of the permit application.

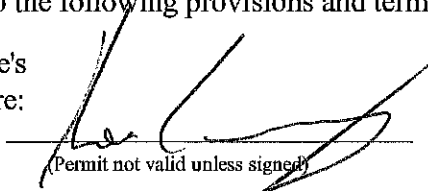
Possession/Transportation of injurious aquatic life species requires appropriate permits in addition to the previously stated. The IDNR Aquaculture Specialist can be contacted to request and seek approval of all project activities of the permit applicant prior to activities being initiated. If such species are encountered as part of previously authorized projects, they may be kept for deposition into state, scientific, educational, or zoological institutions, if appropriate precautions are taken to further restrict potential release into the environment AND immediate reporting of escape to [dnr.aquaculture@illinois.gov](mailto:dnr.aquaculture@illinois.gov). (All aquatic life may be immediately returned unharmed from where they were taken. 515 ILCS 5/10-100.)

Authorization: Statewide, exclusive of nature preserves, and IDNR owned and managed properties

Individuals authorized to work under direct supervision of permittee: None

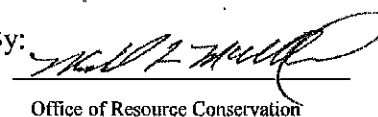
I agree to the following provisions and terms of this Scientific Permit.

Permittee's  
Signature:



(Permit not valid unless signed)

Approved By:



Office of Resource Conservation

Date: 3/4/2022

### TERMS FOR SCIENTIFIC PERMIT

1. This permit is valid only for the approved methods, locations and activities stated on the permit.
2. All permitted activities shall be performed by or under the direct supervision of the permittee. Permittee must be present with persons involved in actual taking of fauna.
3. Under no circumstances shall a scientific permit be used in lieu of sport or commercial licenses.
4. This permit is valid only for species not listed as Illinois Threatened or Endangered (<https://www.dnr.illinois.gov/conservation/NaturalHeritage/Pages/EndangeredandThreatenedSpecies.aspx>). If a Threatened or Endangered species is incidentally captured, the specimen must be released and the occurrence must be reported to [tara.kioninger@illinois.gov](mailto:tara.kioninger@illinois.gov) within 5 business days.
5. This permit does not allow the privilege of trespass. Landowner permission is required. Activities on Department sites are not permitted without the prior approval of the Site Superintendent. Activities on Illinois Nature Preserves and Land and Water Reserves must have prior approval from the Illinois Nature Preserve Commission.
6. Permittee must carry this permit at all times when taking specimens and be presented, upon request, to Department personnel.
7. Fauna taken and/or salvaged and rehabilitated must be released to the wild or permanently donated to a public or state scientific educational or zoological institution.
8. This permit does not supersede Federal permits, which may be necessary for the permitted work.
9. All gear left unattended must be tagged bearing name and scientific permit number of permittee.
10. Use of rotenone or any other toxic materials for taking of fauna must have written approval from the Department prior to using such materials, and may need a variance from the Illinois Environmental Protection Agency.
11. An annual report must be submitted to the Department by January 31 of each year.
12. This permit may be revoked or suspended if the Department finds that a permittee has falsified information on the application, failed to comply with the provisions of this permit, or violated state or federal laws.

The Department of Natural Resources is an equal opportunity employer.

## ILLINOIS DEPARTMENT OF NATURAL RESOURCES

Authorization is hereby granted, under 520 ILCS 5/3.22 and 515 ILCS 5/20-100 of the Illinois Compiled Statutes and 17 ILL. Adm. Code 520 to:

Last Name: **Hilbert**

First Name: **Patrick**

Permit Number: **A22.5491**

Issued: **3/4/2022**

Expires: **12/31/2022**

Business Name/Institution: **EA Engineering, Science, and Technology, I**

Street Address: **444 Lake Cook Rd. Suite 18**

City: **Deerfield, IL**

Zip Code: **60015**

for strictly scientific, educational or zoological purposes, to take the Illinois fauna identified below subject to the following provisions:

May legally capture, by scientifically accepted methods, only the specific aquatic life species listed on the accompanying Illinois Department of Natural Resources (IDNR) scientific permit application/project proposal (on file in Springfield, IL) strictly for scientific, educational, and/or zoological purposes (except endangered and threatened species). After data has been humanely collected from these species, all animals shall be released unharmed at or near the original site of capture. Deceased animals and/or animal parts must be buried or given to a public or state scientific educational or zoological institution. A federal permit is required for all projects involving federally regulated species. If endangered and threatened species are to be taken, the IDNR Division of Natural Heritage, Endangered Species Coordinator must be notified and must approve in writing all project related activities of the permit application.

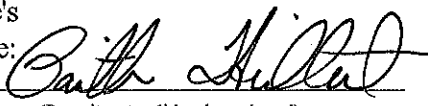
Possession/Transportation of injurious aquatic life species requires appropriate permits in addition to the previously stated. The IDNR Aquaculture Specialist can be contacted to request and seek approval of all project activities of the permit applicant prior to activities being initiated. If such species are encountered as part of previously authorized projects, they may be kept for deposition into state, scientific, educational, or zoological institutions, if appropriate precautions are taken to further restrict potential release into the environment AND immediate reporting of escape to [dnr.aquaculture@illinois.gov](mailto:dnr.aquaculture@illinois.gov). (All aquatic life may be immediately returned unharmed from where they were taken. 515 ILCS 5/10-100.)

Authorization: Statewide, exclusive of nature preserves, and IDNR owned and managed properties

Individuals authorized to work under direct supervision of permittee: None

I agree to the following provisions and terms of this Scientific Permit.

Permittee's

Signature: 

(Permit not valid unless signed)

Approved By: 

Office of Resource Conservation

Date: 3/4/2022

### TERMS FOR SCIENTIFIC PERMIT

1. This permit is valid only for the approved methods, locations and activities stated on the permit.
2. All permitted activities shall be performed by or under the direct supervision of the permittee. Permittee must be present with persons involved in actual taking of fauna.
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4. This permit is valid only for species not listed as Illinois Threatened or Endangered (<https://www.dnr.illinois.gov/conservation/NaturalHeritage/Pages/EndangeredandThreatenedSpecies.aspx>). If a Threatened or Endangered species is incidentally captured, the specimen must be released and the occurrence must be reported to [tara.kieninger@illinois.gov](mailto:tara.kieninger@illinois.gov) within 5 business days.
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12. This permit may be revoked or suspended if the Department finds that a permittee has falsified information on the application, failed to comply with the provisions of this permit, or violated state or federal laws.

The Department of Natural Resources is an equal opportunity employer.

ILLINOIS DEPARTMENT OF NATURAL RESOURCES

Authorization is hereby granted, under 520 ILCS 5/3.22 and 515 ILCS 5/20-100 of the Illinois Compiled Statutes and 17 ILL. Adm. Code 520 to:

Last Name: **Renik** First Name: **Matthew** Permit Number: **A22.6108**

Issued: **3/4/2022**

Expires: **12/31/2022**

Business Name/Institution: EA Engineering, Science, and Technology, I

Street Address: 444 Lake Cook Rd. Suite 18 City: Deerfield, IL Zip Code: 60015

for strictly scientific, educational or zoological purposes, to take the Illinois fauna identified below subject to the following provisions:

May legally capture, by scientifically accepted methods, only the specific aquatic life species listed on the accompanying Illinois Department of Natural Resources (IDNR) scientific permit application/project proposal (on file in Springfield, IL) strictly for scientific, educational, and/or zoological purposes (except endangered and threatened species). After data has been humanely collected from these species, all animals shall be released unharmed at or near the original site of capture. Deceased animals and/or animal parts must be buried or given to a public or state scientific educational or zoological institution. A federal permit is required for all projects involving federally regulated species. If endangered and threatened species are to be taken, the IDNR Division of Natural Heritage, Endangered Species Coordinator must be notified and must approve in writing all project related activities of the permit application.


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Authorization: Statewide, exclusive of nature preserves, and IDNR owned and managed properties

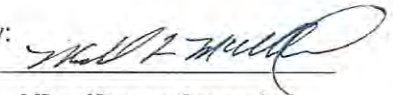
Individuals authorized to work under direct supervision of permittee: None

I agree to the following provisions and terms of this Scientific Permit.

Permittee's

Signature: 

(Permit not valid unless signed)

Approved By: 

Office of Resource Conservation

Date: **3/4/2022**

**TERMS FOR SCIENTIFIC PERMIT**

1. This permit is valid only for the approved methods, locations and activities stated on the permit.
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The Department of Natural Resources is an equal opportunity employer.

ILLINOIS DEPARTMENT OF NATURAL RESOURCES

Authorization is hereby granted, under 520 ILCS 5/3.22 and 515 ILCS 5/20-100 of the Illinois Compiled Statutes and 17 ILL. Adm. Code 520 to:

Last Name: Sneen

First Name: Martin

Permit Number: A22.0500

Issued: 3/4/2022

Expires: 12/31/2022

Business Name/Institution: EA Engineering, Science, and Technology, I

Street Address: 444 Lake Cook Rd. Suite 18

City: Deerfield, IL

Zip Code: 60015

for strictly scientific, educational or zoological purposes, to take the Illinois fauna identified below subject to the following provisions:

May legally capture, by scientifically accepted methods, only the specific aquatic life species listed on the accompanying Illinois Department of Natural Resources (IDNR) scientific permit application/project proposal (on file in Springfield, IL) strictly for scientific, educational, and/or zoological purposes (except endangered and threatened species). After data has been humanely collected from these species, all animals shall be released unharmed at or near the original site of capture. Deceased animals and/or animal parts must be buried or given to a public or state scientific educational or zoological institution. A federal permit is required for all projects involving federally regulated species. If endangered and threatened species are to be taken, the IDNR Division of Natural Heritage, Endangered Species Coordinator must be notified and must approve in writing all project related activities of the permit application.

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Authorization: Statewide, exclusive of nature preserves, and IDNR owned and managed properties

Individuals authorized to work under direct supervision of permittee: None

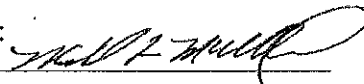
I agree to the following provisions and terms of this Scientific Permit.

Permittee's  
Signature:



(Permit not valid unless signed)

Approved By:



Office of Resource Conservation

Date: 3/4/2022

TERMS FOR SCIENTIFIC PERMIT

1. This permit is valid only for the approved methods, locations and activities stated on the permit.
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5. This permit does not allow the privilege of trespass. Landowner permission is required. Activities on Department sites are not permitted without the prior approval of the Site Superintendent. Activities on Illinois Nature Preserves and Land and Water Reserves must have prior approval from the Illinois Nature Preserve Commission.
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11. An annual report must be submitted to the Department by January 31 of each year.
12. This permit may be revoked or suspended if the Department finds that a permittee has falsified information on the application, failed to comply with the provisions of this permit, or violated state or federal laws.

The Department of Natural Resources is an equal opportunity employer.



# Endangered and Threatened Species Permit

Permit Number: **15074**

Issued Date: 7/5/2022

Expiration Date: 12/31/2022

This permit is valid for the following Counties in Illinois:

- Will
- Grundy
- Crawford

Pursuant to 520 ILCS 10/5 and 17 Ill. Adm. Code 1070.10-1070.80, this permit is issued to:

**Larry Bushing**  
**444 Lake Cook Rd. Suite 18**  
**Deerfield, IL 60015**

and covers the following additional personnel:

- Joe Vondruska
- Katelyn Jackson
- Ken Cummings
- Mike Kacinski
- Patrick Hilbert
- Marty Sneen
- Matt Renik

from:

**EA Engineering, Science, and Technology, Inc., PBC**

for the purpose of SCIENTIFIC RESEARCH involving the following specimens and/or products:

<i>Species</i>	<i>Item</i>	<i># Specimens/ Products</i>	<i>Collection Method</i>	<i>Action</i>	<i>Disposition</i>
Fish - Pallid Shiner - <i>Hybopsis amnis</i>	Live Individual	All captured	Nets or Seines	Observe	Catch and Release Live Specimen
Fish - Pallid Shiner - <i>Hybopsis amnis</i>	Live Individual	All captured	Electrofishing	Observe	Catch and Release Live Specimen
Fish - Pallid Shiner - <i>Hybopsis amnis</i>	Live Individual	All difficult to ID specimens	Nets or Seines	Lethal Take	Lethal Take
Fish - Pallid Shiner - <i>Hybopsis amnis</i>	Live Individual	All difficult to ID specimens	Electrofishing	Lethal Take	Lethal Take
Fish - Greater Redhorse - <i>Moxostoma valenciennesi</i>	Live Individual	All captured	Nets or Seines	Observe	Catch and Release Live Specimen
Fish - Greater Redhorse - <i>Moxostoma valenciennesi</i>	Live Individual	All captured	Electrofishing	Observe	Catch and Release Live Specimen
Fish - Greater Redhorse - <i>Moxostoma valenciennesi</i>	Live Individual	All difficult to ID specimens	Nets or Seines	Lethal Take	Lethal Take

Electronic Filing: Received, Clerk's Office 10/6/2023

Fish - Greater Redhorse - <i>Moxostoma valenciennesi</i>	Live Individual	All difficult to ID specimens	Electrofishing	Lethal Take	Lethal Take
Fish - River Redhorse - <i>Moxostoma carinatum</i>	Live Individual	All captured	Nets or Seines	Observe	Catch and Release Live Specimen
Fish - River Redhorse - <i>Moxostoma carinatum</i>	Live Individual	All captured	Electrofishing	Observe	Catch and Release Live Specimen
Fish - River Redhorse - <i>Moxostoma carinatum</i>	Live Individual	All difficult to ID specimens	Nets or Seines	Lethal Take	Lethal Take
Fish - River Redhorse - <i>Moxostoma carinatum</i>	Live Individual	All difficult to ID specimens	Electrofishing	Lethal Take	Lethal Take
Fish - American Eel - <i>Anguilla rostrata</i>	Live Individual	All captured	Nets or Seines	Observe	Catch and Release Live Specimen
Fish - American Eel - <i>Anguilla rostrata</i>	Live Individual	All captured	Electrofishing	Observe	Catch and Release Live Specimen
Fish - American Eel - <i>Anguilla rostrata</i>	Live Individual	All difficult to ID specimens	Nets or Seines	Lethal Take	Lethal Take
Fish - American Eel - <i>Anguilla rostrata</i>	Live Individual	All difficult to ID specimens	Electrofishing	Lethal Take	Lethal Take
Fish - Blackchin Shiner - <i>Notropis heterodon</i>	Live Individual	All captured	Nets or Seines	Observe	Catch and Release Live Specimen
Fish - Blackchin Shiner - <i>Notropis heterodon</i>	Live Individual	All captured	Electrofishing	Observe	Catch and Release Live Specimen
Fish - Blackchin Shiner - <i>Notropis heterodon</i>	Live Individual	All difficult to ID specimens	Nets or Seines	Lethal Take	Lethal Take
Fish - Blackchin Shiner - <i>Notropis heterodon</i>	Live Individual	All difficult to ID specimens	Electrofishing	Lethal Take	Lethal Take
Fish - Western Banded Killifish - <i>Fundulus diaphanus menona</i>	Live Individual	All captured	Nets or Seines	Observe	Catch and Release Live Specimen
Fish - Western Banded Killifish - <i>Fundulus diaphanus menona</i>	Live Individual	All captured	Electrofishing	Observe	Catch and Release Live Specimen
Fish - Western Banded Killifish - <i>Fundulus diaphanus menona</i>	Live Individual	All difficult to ID specimens	Nets or Seines	Lethal Take	Lethal Take
Fish - Western Banded Killifish - <i>Fundulus diaphanus menona</i>	Live Individual	All difficult to ID specimens	Electrofishing	Lethal Take	Lethal Take
Fish - Bigeye Chub - <i>Hybopsis amblops</i>	Live Individual	All captured	Nets or Seines	Observe	Catch and Release Live Specimen
Fish - Bigeye Chub - <i>Hybopsis amblops</i>	Live Individual	All captured	Electrofishing	Observe	Catch and Release Live Specimen

If the research project covered by this permit will involve propagation, the permit holder and additional personnel listed above are required to possess an IDNR endangered and threatened species permit Propagation Addendum.

Possession of federally listed species is covered by:

Questions about this permit should be directed to [DNR.ETPermit@Illinois.gov](mailto:DNR.ETPermit@Illinois.gov)

**USDA Exhibitor Permit #**

**U.S. Fish and Wildlife Service Permit #**


The research project covered by this permit will address:

- |                                                                                            |                                                                                                      |
|--------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------|
| <input checked="" type="checkbox"/> <i>Distribution or status of the listed species</i>    | <input type="checkbox"/> <i>Threats to the listed plants and animals and/or their habitats</i>       |
| <input type="checkbox"/> <i>Life history of the listed species</i>                         | <input type="checkbox"/> <i>Effects of exotic species on native populations</i>                      |
| <input type="checkbox"/> <i>Ecological needs of the natural populations of the species</i> | <input type="checkbox"/> <i>Genetic diversity within population</i>                                  |
| <input type="checkbox"/> <i>Supplementing existing populations</i>                         | <input type="checkbox"/> <i>Wildlife disease vectors and transmission</i>                            |
| <input type="checkbox"/> <i>Captive rearing</i>                                            | <input type="checkbox"/> <i>Translocation to unoccupied locations within species' historic range</i> |
| <input type="checkbox"/> <i>Effects of management actions on animals or plants</i>         | <input type="checkbox"/> <i>Impact of wind turbines on listed species</i>                            |
| <input type="checkbox"/> <i>Movement or habitat use</i>                                    | <input type="checkbox"/> <i>Propagation for release into the wild</i>                                |
| <input type="checkbox"/> <i>Other:</i>                                                     |                                                                                                      |

The specific locations where this research will be conducted are:

<i>Research Location</i>	<i>Nearest City</i>
Chicago Sanitary and Ship Canal	Lockport
Des Plaines River	Joliet and Channahon
Kankakee River	Lorenzo
Illinois River	Lorenzo
Robinson Creek	Robinson

**ITEMS LISTED ON THIS PERMIT MAY BE SOLD,  
GIVEN AWAY, OR OTHERWISE DISPOSED OF ONLY  
WITH PERMISSION OF THE ILLINOIS  
DEPARTMENT OF NATURAL RESOURCES.**

Signed:   
**Christopher Young**  
Office Director  
IDNR Office of Resource Conservation  
*As designee of IDNR Director, Wayne A. Rosenthal*

Special Conditions (IF APPLICABLE):

**Before any research is conducted within an Illinois DNR site, permission from the Site Superintendent must be granted. Research within a Nature Preserve or Land and Water Reserve cannot occur unless written authorization/special use permit is received from the Illinois Nature Preserves Commission.**

**These surveys are in compliance with Midwest Generation's thermal effluent permit from the Pollution Control Board. Surveys have been conducted 1997.**

**The Illinois DNR strongly recommends limiting the number of vouchers as a Special Condition of this permit. The IDNR strongly recommends that wherever possible, one (1) individual of each listed species from each survey location be vouchered.**

**Please note that any movement/translocation of any and all listed species within the State of Illinois is prohibited unless such activities are specifically covered under an official, approved IDNR Incidental Take Authorization (ITA). Without an ITA, all animals shall be returned unharmed at or near their original capture/discovery location**

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immediately after photographing the specimen(s), recording location information, and data has been humanely collected if applicable. The Department shall be notified within 48 hours of discovery of any such listed species. Please contact Joe Kath via email with such information: Joe.Kath@illinois.gov

Conditions:

- A copy of this permit must be in the possession of the permit holder when engaged in activities involving endangered or threatened species.
- There shall be no propagation of or attempt to propagate any endangered or threatened species covered by this permit unless a signed IDNR addendum approving propagation is attached. In addition, the Propagation Addendum must be in the possession of the permit holder when engaged in all activities involving propagation of an Illinois listed species.
- Permit holder cannot move/transport/translocate any endangered or threatened species outside of a designated project area/zone of impact without expressed written consent of the Director of the Illinois Department of Natural Resources.
- Permit holder shall notify IDNR of any changes to personal information within 10 days of making such changes.
- Permit holder shall notify IDNR of any changes to inventory of specimens through escape, theft, death or other unanticipated events within five working days of the discovery of loss.
- Permit holder must provide the Department with an electric copy or two hard copies of any reports, technical papers, or technical notes that result from studies conducted under the auspices of this permit.
- An annual report must be submitted to IDNR by January 31st of each year.

The holder of this permit may:

- Dispose of specimens or products covered by this permit through transfer or scrapping only after a permit/written permission has been applied for and received from the Department.
- Allow temporary possession of the items covered by this permit by a licensed taxidermist for the purpose of providing taxidermic services.

**This permit may be revoked if the Department finds that a permittee has falsified information on the application, failed to comply with facilities standard or animal welfare standards established in 17 Ill. Adm. Code 1070.60 and 1070.70, or violated state or federal laws**



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**Appendix E**  
**Fish Sampling Data Sheet**

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Data ID #  Project #  Project Name  Site

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Gear  Location  Sampling Effort  Page \_\_\_ of \_\_\_

Start Datetime  :  :   
D D M M M Y Y h h m m

End Datetime  :  :   
D D M M M Y Y h h m m

**Water Quality Information**Serial Number: 

Depth	Temp (C)	D.O.					<b>Field Notes</b>	
								<input type="checkbox"/> Specimens preserved (X)

OUTPUT	
volts	<input type="text"/>
amps	<input type="text"/>

Common Name	SPCODE	L S	Length	Weight	Plus Count	Batch Weight	Anomalies					REMARKS
							A1	A2	A3	A4	<1"	

1. Body deformity  
2. Eroded fins  
3. Lesions-Ulcers  
4. Tumors

5. Anchor worm-light  
6. Anchor worm-heavy  
7. Blackspot-light  
8. Blackspot-heavy

9. Leeches-light  
10. Leeches-heavy  
11. Fungus  
12. Ich

13. Blind  
14. Emaciated  
15. External parasites-other  
16. Popeye

17. Swirled scales  
18. Other  
< 1"(25cm) = 99  
Life Stage (LS): YOY=6



**Appendix F**  
**Multiprobe Water Quality Monitoring Instruments**

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**Standard Operating Procedure No. 043  
for Multi-Probe Water  
Quality Monitoring Instruments**

*Prepared by*

EA Engineering, Science, and Technology, Inc., PBC  
225 Schilling Circle, Suite 400  
Hunt Valley, Maryland 21031

Revision No. 2  
June 2020



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### PROJECT-SPECIFIC VARIANCE FORM

This form is to be completed to indicate if there are any client-, project-, or site-specific variances to this Standard Operating Procedure (SOP) (**also check Box A**), or if this SOP is being used with no changes (**only check Box B**).

- A. Variances required; cite section(s) of the SOP to which there is a variance**
- B. No variances**

SOP No. 043	
SOP Section	Variance

---

Project Manager (Name)

---

Project Manager (Signature) Date

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ATTACHMENT A: EQUIPMENT CALIBRATION LOG

DOCUMENT REVISION HISTORY

<b>ORIGINAL (MASTER) DOCUMENT REVISION HISTORY</b>				
<b>Revision Number</b>	<b>Revision Date</b>	<b>Revision Summary</b>	<b>Revised By</b>	<b>Reviewed By</b>
2	June 2020	Systematic review and update. Combined with previously separate SOPs for pH, temperature, specific conductivity, turbidity, dissolved oxygen, and redox potential.	Eddie Meadows Catherine Maxwell	Matthew Bowman

## 1. PURPOSE AND SCOPE

The purpose of this Standard Operating Procedure (SOP) is to delineate protocols for field operation of multi-probe water quality instruments. The instrument can monitor a variety of basic parameters including dissolved oxygen, percent saturation, temperature, pH, conductivity, specific conductivity, resistivity, salinity, total dissolved solids, turbidity, oxidation reduction potential (ORP), level, and depth.

The use of brand names in this SOP is not intended as endorsement or mandate that a given brand be used. Alternate equivalent brands of detectors, sensors, meters, etc. are acceptable. If alternate equipment is to be used, the vendor must provide applicable and comparable SOPs for the maintenance and calibration from the specific manufacturer of the instrument being used.

## 2. MATERIALS

The following materials may be required:

- Multi-probe instrument
- Probe/sonde with appropriate cables
- Appropriate standards/calibration fluids
- Accessories (batteries, charger, case, etc.)
- Decontamination materials or laboratory wipes
- Deionized water and distilled water (as needed for calibration and decontamination)
- Instrument logbook
- Manufacturer's Operations Manual.

## 3. CALIBRATION PROCEDURE

Calibration must be performed or verified daily at a minimum before using the instrument. Calibration may be performed in the laboratory or in the field. Detailed step-by-step calibration procedures for the equipment described below are provided in the most recent version of the manufacturer's Operations Manual. Documentation includes at a minimum: time, date, analyst, standard, primary standard/calibration fluid lot number, secondary standard/calibration fluid lot number, and expiration dates of standards/calibration fluids. An example calibration log is provided in Attachment A.

Fill the calibration cup with the appropriate standard as follows:

- Temperature: None required
- Specific Conductance: Conductivity standards
- pH: pH 7 buffer plus pH 4 and/or pH 10 buffer
- Dissolved Oxygen: Saturated air or saturated water
- ORP: Quinhydrone (Zobell's Solution) or other standard

- Turbidity: Nephelometric turbidity unit (NTU) standards
- Salinity: Calibration for specific conductance
- Depth/Level: Set zero in air.

### 3.1 CONDUCTIVITY CALIBRATION

Conductivity meters are calibrated at least once per day to at least one standard. The standard should be selected in accordance with the range expected to be measured (e.g., 1.0 microSiemens per centimeter [ $\mu\text{S}/\text{cm}$ ]) standard should not be used to calibrate meters being used in saltwater). See manufacturer's recommendations in the Operations Manual for additional information on calibration standard selection. Calibration information is recorded in conjunction with the data collected for that sampling event.

### 3.2 PH CALIBRATION

The pH meters are calibrated at least once per day to a minimum of two standard buffers (pH 4 and 7, or pH 7 and 10) in accordance with the range expected to be measured. The calibration is verified using a fresh solution of pH 7 buffer post-calibration. The probe should be rinsed in distilled water between standards. Calibration information is recorded in conjunction with the data collected for that sampling event.

### 3.3 DISSOLVED OXYGEN CALIBRATION

Dissolved oxygen meters are saturated-air or saturated-water calibrated at least once per day. Each method requires the true barometric pressure to be input or collected from the instrument prior to calibration.

- ***Saturated Air Method***—Dip the calibration chamber (i.e., probe storage cup) into distilled or tap water at ambient temperature, pour out excess water, and then insert dissolved oxygen probe into the wet chamber. This ensures that the air inside the chamber is saturated with water vapor. CAUTION: Be sure that the membrane/probe has no droplets of water adhering to it since this would reduce the rate of oxygen diffusion through the membrane and would produce erroneous results. Do not fully thread the probe storage cup on the probe during equilibration.
- ***Saturated Water Method***—To make a 100 percent (%) air-saturated calibration standard, fill a container (e.g., a 1-liter or 1-gallon container with a closed top) three-quarters full with distilled water or clean (conductivity of less than  $500 \mu\text{S}/\text{cm}$ ) tap water. Let the water temperature reach equilibrium with the calibration environment. Then shake the container vigorously for approximately 30 seconds. This makes 100% air-saturated water. Place the air-saturated water into the probe storage cup and allow to equilibrate. Do not fully thread the probe storage cup on the probe during equilibration.

Calibration information is recorded in conjunction with the data collected for that sampling event.



### **3.4 OXIDATION REDUCTION POTENTIAL CALIBRATION**

ORP meters are calibrated at least once per day to at least one standard. It is recommended that Zobell's Solution is used; however, another solution can be used as long as it meets the manufacturer's specifications for calibration. Note that the standard value for Zobell's Solution is dependent on temperature. Calibration information is recorded in conjunction with the data collected for that sampling event.

### **3.5 TURBIDITY CALIBRATION**

The turbidity meters are calibrated at least once per day to a minimum of two standards (0 NTU and 100 or 200 NTUs recommended) in accordance with the range expected to be measured. Calibration information is recorded in conjunction with the data collected for that sampling event.

### **3.6 DEPTH/LEVEL CALIBRATION**

The depth and level calibration is performed with the depth sensor module in the air and not immersed in any solution. The appropriate correction for height above the water surface is inputted into the meter. Calibration information is recorded in conjunction with the data collected for that sampling event.

### **3.7 ADDITIONAL CALIBRATIONS**

Additional measurements may be taken with the multi-probe water quality instruments. For any of these measurements, the calibration procedures will be conducted in accordance with the manufacturer's specifications. Calibration information is recorded in conjunction with the data collected for that sampling event.

## **4. FIELD OPERATION**

### **4.1 SETUP OF MULTI-PROBE WATER QUALITY INSTRUMENT**

Post-calibration and prior to sampling, the multi-probe water quality instrument should be inspected, cleaned, and set up for data collection. If the cables have been unattached, they will be reconnected to the transmitter (if applicable) and the display. Once all cables are attached, the meter will be turned on and allowed to warm up for a few seconds in order to allow the display screen to load. The unit should be allowed to come to ambient air temperature if it has been stored in a hotter or colder environment prior to use.

### **4.2 SURFACE WATER**



Prior to sampling, check the condition of the probes before each deployment. When sampling in surface water, the sensor must be in an amount of water sufficient for all probes to be submerged. Data values displayed on the display screen are recorded in a field logbook, a dedicated project field form (i.e., an EA Purging and Sampling Record, or on an EA-provided iPad on an approved GoFormz), and accepted into the instrument's data logger (if used). Post-data collection, the sensor will be retrieved and rinsed for use at the next sample location. If travel time between sample locations is significant, the display is to be turned off. When all sampling is completed, disconnect all equipment, clean probes and the instrument in accordance with the manufacturer's instructions, attach a solid protective cap, and return it to its proper storage location.

### **4.3 GROUNDWATER**

Prior to sampling, check the condition of the probes before each deployment. When sampling groundwater, mount sampler on a flow-through cell. Start sampler pump and allow pump/hose system to be purged of air bubbles. Required parameters should be recorded every 3-5 minutes (unless otherwise specified in the sampling plan). Record the monitored values in the appropriate field logbook, on a dedicated project field form (i.e., an EA Purging and Sampling Record, or on an EA provided iPad on an approved GoFormz) to ensure against inadvertent data loss. If travel time between sample locations is significant, the display is to be turned off. When all sampling is completed, disconnect all equipment, clean probes and the instrument in accordance with the manufacturer's instruction, remove flow-through cell and attach solid protective cap, and return it to its proper storage location. If a flow-through cell cannot be used (e.g., groundwater sampling using a bailer), bailed water should be poured into a clean container for collecting readings over standard intervals of volume purged or time.

## **5. MAINTENANCE**

All maintenance should be performed in accordance with the manufacturer's Operations Manual.

## **6. PRECAUTIONS**

Check the condition of the probes frequently between sampling. Do not force pins into connections; note keying sequence. If field readings are outside the expected range, check for bubbles on, or damage to, the probes. If there are no bubbles or damage, recalibrate the sensor.

## **7. REFERENCES**

Not applicable.

# **Attachment A**

## **Equipment Calibration Log**



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EA Engineering, Science, and Technology, Inc., PBC



**ATTACHMENT A**  
**EQUIPMENT CALIBRATION LOG**

<b>Site Name:</b>		<b>Client:</b>	
<b>Job Number:</b>		<b>Calibration Performed by:</b>	Page _____ of _____

Date and Time	Instrument Name	Instrument Make and Model	Instrument Serial Number	Standard Value	Calibrated Value	Percent Deviation	Bump Check (if applicable)	Standard/ Calibration Fluid Lot Number and Expiration Date	Comments or Adjustments Made



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**ATTACHMENT B**

**Record of Calibration/Checking:  
Temperature, DO Meter, Conductivity, pH**

Client: \_\_\_\_\_ Instrument Model (Primary): \_\_\_\_\_  
 Project Number: \_\_\_\_\_ Primary Unit-Serial #: \_\_\_\_\_  
 Sampling Date: \_\_\_\_\_ Instrument Model (Backup): \_\_\_\_\_  
 Calibrated By and Date: \_\_\_\_\_ Backup Unit-Serial #: \_\_\_\_\_

Site (Circle One): LAB / FIELD	Temperature( °C)		Dissolved Oxygen (mg/L)		Specific Conductance(µS)
Standard Used: (Circle One)	USB Thermometer/ Meter Comparison		Winkler / Meter Comparison		Conductivity Standard / Meter Comparison
Instrument Reading in Water Bath:					***Meter Reading in Conductivity Standard:
Standard Reading <sup>1</sup> :					Conductivity Standard = 1000 µS
Post Calibration Reading:	X				
Instrument Adjustment:					
<b>REMARKS:</b>					
<sup>1</sup> Traceable Thermometer and Winkler Reading of Water Bath. Connectivity reading in Standard Solution					

**pH:**

Meter: (Serial#)	Primary:			Backup:			pH Pen:		
<b>pH Standards Used</b> (Circle All Used):	4	7	10	4	7	10	4	7	10
Instrument Reading in Water Bath:									
Instrument Reading in Standard:									
Post-Cal. Reading in Water Bath:									
Instrument Adjustment:									
<b>REMARKS:</b>									

**pH Calibration:** 1. Record meter readings of water bath. 2. Record reading of meters in pH Buffer Solutions then calibrate to the standard. 3. Record post-cal. reading of meters in water bath.

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# Professional *Plus*



## Calibration Tips



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## **Introduction**

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This guide provides helpful instructions, tips and troubleshooting suggestions for calibrating a Professional Plus instrument. For more detailed information on calibration and information on how to setup and operate a Pro Plus, please refer to the Pro Plus User Manual.

## **Calibration Worksheet**

---

The Calibration Worksheet on the following page is provided for your convenience. Utilizing the Calibration Worksheet can help document your calibration and track the performance of your sensors.

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Date of Calibration: \_\_\_\_\_ Technician: \_\_\_\_\_  
 Instrument Serial Number: \_\_\_\_\_ Software Revision: \_\_\_\_\_ Cable Model Number: \_\_\_\_\_  
 Temperature Reading \_\_\_\_\_ Temperature Accurate: Y N  
 DO Sensor in use: Polarographic Galvanic Sensor notated in Sensor menu? Y N  
 DO membrane changed? Y N Color of Membrane \_\_\_\_\_ Color notated in Sensor menu? Y N

Record the following calibration values:

	Pre Cal	After Cal	
Conductivity	_____	_____	
ORP	_____	_____	
DO	_____	_____	True Barometric Pressure at time of calibration _____

	Pre Cal		
pH 7	_____	pH mV value _____	Range 0 mV ± 50 mV
pH 4	_____	pH mV value _____	Range +165 to +180 from 7 buffer mV value
pH 10	_____	pH mV value _____	Range -165 to -180 from 7 buffer mV value

NOTE: See pH Cal tips section for additional information. Span between pH 4 and 7 and 7 and 10 mV values should be ≈ 165 to 180 mV. 177 is the ideal distance or 59 mV per pH unit.

**Ammonium**  
 1<sup>st</sup> point (1 mg/L) \_\_\_\_\_ NH4 mV value \_\_\_\_\_ Range: 0 mV +/- 20 mV (new sensor only)  
 2<sup>nd</sup> point (100 mg/L) \_\_\_\_\_ NH4 mV value \_\_\_\_\_ Range: 90 to 130 mV > 1 mg/L mV value

**Nitrate**  
 1<sup>st</sup> point (1 mg/L) \_\_\_\_\_ NO3 mV value \_\_\_\_\_ Range: 200 mV +/- 20 mV (new sensor only)  
 2<sup>nd</sup> point (100 mg/L) \_\_\_\_\_ NO3 mV value \_\_\_\_\_ Range: 90 to 130 mV < 1 mg/L mV value

**Chloride**  
 1<sup>st</sup> point (10 mg/L) \_\_\_\_\_ Cl mV value \_\_\_\_\_ Range: 225 mV +/- 20 mV (new sensor only)  
 2<sup>nd</sup> point (1000mg/L) \_\_\_\_\_ Cl mV value \_\_\_\_\_ Range: 80 to 130 < 10 mg/L mV value

Record the following diagnostic numbers **after** calibration, by viewing the .glp file and reading the values for the day's calibration

Conductivity Cal Cell Constant \_\_\_\_\_ Range 5.0 +/- 1.0 acceptable  
 DO Sensor Value (uA) \_\_\_\_\_ (Membrane dependent, see DO Cal Tips)  
 pH Slope \_\_\_\_\_ (≈ 55 to 60 mV/pH, 59 ideal)  
 pH Slope % of ideal \_\_\_\_\_

## Temperature

---

### CALIBRATION TIPS

Before calibrating any other Pro Plus sensor, verify that the temperature sensor is reading accurately by comparing it to a traceable thermometer or other known reference in a water bath. Temperature compensation is used in every other sensor measurement so its accuracy should be verified and recorded each time the Pro Plus is calibrated. Be sure to consider the specification tolerances of both the Pro Plus temperature sensor and the thermometer when comparing the measurements.

The Pro Plus temperature sensor can not be calibrated nor should calibration be required.

### TROUBLESHOOTING TIPS

If the temperature sensor is not reading accurately, ensure that it is clean and free of debris. The conductivity cleaning brush and warm water with mild detergent can be used to scrub the temperature sensor if needed. Alternatively, you can use a toothbrush to clean the sensor.

### Quatro Cables

Quatro cables have a replaceable combination conductivity/temperature sensor (p/n 005560). All other Pro Plus cables have integral temperature sensors. If using a Quatro cable and your temperature sensor is not reading accurately, remove the conductivity/temperature sensor from the cable. The Pro Plus should read ----- °C without a temperature sensor installed. If the instrument is reading any other value, the conductivity/temperature port on the cable may be contaminated. Refer to the Cleaning the Sensor Port section of this document for information on how to clean the port.

After cleaning the port, recheck the temperature reading. If the temperature reading is still not displaying ----- °C without the sensor installed, there may be a problem with the cable and/or instrument. In this case, contact your local YSI Representative or a YSI Authorized Service Center.

### Other Pro Plus Cables

If your temperature sensor is not reading accurately after cleaning around the sensor, contact your local YSI Representative or an YSI Authorized Service Center.

## Conductivity

---

The conductivity calibration should be verified every day the instrument is used. However, the conductivity sensor is very stable and may hold its calibration for several weeks.

### CALIBRATION TIPS

1. It is not necessary to calibrate conductivity, specific conductance and salinity. Calibrating one of these parameters will simultaneously calibrate the others. YSI recommends calibrating specific conductance (temperature compensated conductivity) for greatest ease and accuracy.
2. Ensure the conductivity sensor is clean and dry before performing a specific conductance calibration.
3. Always use fresh, traceable conductivity calibration solution when calibrating the conductivity sensor.
  - a. The shelf life of conductivity solution is one month after being opened. This is due to potential changes in the value of the solution caused by evaporation which can occur after opening the bottle. Be sure to write the open date on the bottle so you know that you are using good calibration solution.
  - b. Never calibrate with a conductivity solution that is less than 1.0 mS/cm. You are setting the slope on a linear device so a good strong conductivity signal will give you the best performance. Use 1.0 mS/cm for fresh water, 10 mS/cm for brackish to estuarine water and 50 mS/cm for salt water. 1.0 mS (millisiemens) = 1000 uS (microsiemens).
4. Pre-rinse the cal cup and sensors with a small amount of calibration standard or rinse standard and discard.
5. When calibrating the conductivity sensor, the calibration solution must cover the top vent holes of the conductivity sensor. If using a Quatro cable, the top vent hole is located on the side of the combination conductivity/temperature sensor. If using a different cable, the conductivity sensor is integral to the cable and the sensor has two vent holes located close to the cable. Ensure the entire conductivity sensor is submerged in the solution or the instrument will read approximately half the expected value.
6. After placing the sensor into the solution, gently move the sensor up and down to remove any air bubbles that may be trapped in the conductivity sensor.
7. If calibrating Specific Conductance, enter the value of the conductivity solution as it is listed for 25°C. Make sure you are entering the correct units. 1 mS = 1,000 uS.
8. If you receive a warning message stating that the calibration is questionable, do not continue with the calibration. Instead, select 'No' and investigate what is causing the questionable results. If you accept a questionable calibration, your conductivity readings (and your DO mg/L readings) will be erroneous. Typical causes for this error message include: incorrect entries (entering 1000 uS/cm instead of 1.0 mS/cm), not using enough solution to cover the vent holes, air bubbles trapped in the sensor, calibrating in conductivity instead of specific conductance, dirty conductivity electrodes, and/or bad calibration solution.
9. After accepting a good calibration, navigate to the GLP file and check the conductivity cell constant for the calibration. For highest accuracy, the cell constant should be 5.0 +/- 0.5. However, the acceptable range is 5 +/- 1.0. A cell constant outside of this range indicates that a questionable calibration was accepted.

### TROUBLESHOOTING TIPS

If you get an error message during calibration, be sure that you are:

1. Entering the correct calibration value (1 mS/cm = 1000 uS/cm).
2. Calibrating in Specific Conductance mode.
3. Using enough solution to cover the vent holes on the sensor.
4. Dislodging any air bubbles that could be trapped in the sensor.
5. Using a fresh, traceable conductivity calibration solution.

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If you are following the above recommendations and still receiving an error message, check the conductivity sensor to make sure it is clean. A clean conductivity sensor should read less than 3 uS/cm in dry air. If your sensor is dry and giving you a reading higher than 3 uS/cm in air, it should be cleaned.

The conductivity calibration generates its cell constant value after calibration. The ideal cell constant is 5.0 +/-0.5 but 5.0 +/- 1.0 is acceptable. Any significant jump or change in this number from one calibration to the next usually indicates a problem with the calibration and/or sensor. If you are sure that your calibration standard is good and your calibration process is correct, then your sensor may need to be cleaned.

### **Cleaning the Conductivity Sensor**

The openings that allow sample access to the conductivity electrodes should be cleaned regularly. The small cleaning brush included in the Maintenance Kit is intended for this purpose. Dip the brush in clean water and insert it into each hole 10 to 12 times. In the event that deposits have formed on the electrodes, it may be necessary to use a mild detergent (laboratory grade soap or bathroom foaming tile cleaner) with the brush. Rinse thoroughly with clean water, then check the response and accuracy of the conductivity sensor with calibration solution.

### **Quatro Cables**

Quatro cables have a replaceable combination conductivity/temperature sensor (p/n 5560). All other Pro Plus cables have integral conductivity sensors. If using a Quatro cable and your conductivity sensor is not calibrating or is reading > 3 uS/cm in dry air after being cleaned, remove the conductivity/temperature sensor from the cable. The Pro Plus should read < 3 uS/cm for conductivity (not specific conductance) without a conductivity sensor installed. If the instrument is reading > 3 uS/cm without a sensor installed, the conductivity/temperature port on the cable may be contaminated. Refer to the Cleaning the Sensor Port section of this document for information on how to clean the port.

If the conductivity measurement continues to read more than 3 uS/cm without a conductivity/temperature sensor installed, there may be a problem with the cable and/or instrument. In this case, contact your local YSI Representative or a YSI Authorized Service Center.


### **Other Pro Plus Cables**

If your conductivity sensor is not calibrating or is reading > 3 uS/cm in dry air after performing a sensor cleaning, contact your local YSI Representative or a YSI Authorized Service Center.

## pH

The pH calibration should be verified every day the instrument is used. However, a new pH sensor may be capable of holding its calibration for several days.

### CALIBRATION TIPS

1. If using a pH sensor in a 6051010 or Quatro cable, calibrate the sensor in port 1 prior to calibrating the sensor in port 2. The sensor in port 2 uses the reference of the sensor installed in port 1. Therefore, it is important to verify that the port 1 sensor is working properly before calibrating the port 2 sensor. See pH Troubleshooting Tips for additional info.
2. The pH sensor can be calibrated with up to six calibration points.
3. Calibration can be accomplished in any buffer order.
4. pH 7 buffer should be used regardless of how many calibration points you use; however, it does not have to be the first point.
5. In most cases, a two-point calibration is all that is required (4 and 7 or 7 and 10). You can bracket the expected in-situ pH values. Use a three-point calibration with 4, 7 and 10 if the in-situ pH values are unknown or if you expect the in-situ values to be on both sides of the pH scale.
6. Rinse the sensors and cal cup with a small amount of pH buffer. Fill the cup so that the pH sensor tip and the temperature sensor are submerged in buffer.
7. If necessary, highlight the Calibration Value and enter the pH value of the buffer solution. Note: The Pro Plus has auto buffer recognition which can be set to USA (4, 7, 10) or NIST (4.01, 6.86, 9.18) buffer values in the pH Sensor Setup menu.
8. Record the pH millivolts for each calibration point. The acceptable mV outputs for each buffer are shown below.
  - pH 7 mV value = 0 mV +/- 50 mV
  - pH 4 mV value = +165 to +180 from 7 buffer mV value
  - pH 10 mV value = -165 to -180 from 7 buffer mV value
  - A value of +50 or -50 mVs in buffer 7 does not indicate a bad sensor.
  - The mV span between pH 4 and 7 and 7 and 10 mV values should be  $\approx$  165 to 180 mV. 177 is the ideal distance. The slope can be 55 to 60 mV per pH unit with an ideal of 59 mV per pH unit.
  - If the mV span between pH 4 and 7 or 7 and 10 drops below 160, clean the sensor and try to recalibrate.
9. Wait for the pH to stabilize in the each buffer and then press enter to accept each calibration point.
10. Rinse the sensor and cal cup with a small amount of the next buffer between calibration points.
11. After pressing enter to accept your last calibration point, press cal  to complete the calibration. Otherwise you will continue calibrating up to 6 calibration points.
12. If you receive a warning message stating that the calibration is questionable, do not continue with the calibration. Instead, select 'No' and investigate what is causing the questionable results. If you accept a questionable calibration, your pH readings will be erroneous. Typical causes for this error message include: incorrect Sensor/Port setup in the instrument, a dirty sensor or bad buffer solution.
13. After accepting a good calibration, navigate to the GLP file and check the pH Slope and Slope % of ideal. A good slope should be between 55 and 60 mVs while the ideal is 59 mV. If the slope drops below 53, the sensor should be reconditioned and recalibrated.

### TROUBLESHOOTING TIPS

Typical working life for pH sensors is approximately 12-24 months depending on usage, storage and maintenance. Proper storage and maintenance generally extends the sensor's working life.

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Clean and recondition the sensor if a slow response in the field has been reported or if it takes more than 90 seconds to stabilize in pH buffer.

If you get an error message during a pH calibration, check the following:

1. Ensure the pH buffers are good and not expired
2. Ensure that the pH sensor is installed in the correct port of the cable and the correct ISE is enabled in the Sensor Setup menu.
  - a. If using a pH or pH/ORP combo sensor in a 6051020 cable, ensure the sensor is installed in port 1.
  - b. If using a pH or pH/ORP combo sensor in a 60510, 6051020 or 6051030 cable, pH should be enabled in ISE1 of the instrument's Sensor Setup menu.
  - c. If using a pH sensor in a 6051010 or Quatro cable, check to see if the pH sensor is installed in port 1 or port 2. If the pH sensor is installed in port 1, enable pH in ISE1 of the Sensor Setup menu. If the pH sensor is installed in port 2, enable pH in ISE2 of the Sensor Setup menu. Note: It is not recommended to use a pH/ORP combo sensor in 6051010 or Quatro cables. If using a pH/ORP combo sensor in a 6051010 or Quatro cable, ORP will not be measured or reported.
3. If using a 6051010 or Quatro cable, you must have a sensor installed in port 1 for port 2 to operate. Additionally, ensure that the sensor installed in port 1 is in good working order. In 6051010 and Quatro cables, the sensors installed in port 1 and port 2 use the reference from the sensor installed in port 1 only. Therefore, if the sensor installed in port 1 is not working properly, the readings from the sensor installed in port 2 will be erroneous. For greatest ease, install a pH sensor in port 1 of both 6051010 and Quatro cables and your other ISE sensor in port 2.
4. If you continue to get error messages during calibration, clean and recondition the sensor.

### **Cleaning and Reconditioning the pH, ORP or pH/ORP Sensor**

If the pH or pH/ORP sensor has been allowed to dry out or has been stored in distilled or deionized water for an extended period of time, soak the sensor in buffer 4 overnight to try and restore functionality.

Cleaning is required whenever deposits or contaminants appear on the glass and/or platinum surfaces or when the sensor's response slows. The cleaning can be chemical and/or mechanical.

Removing the sensor from the cable may make cleaning easier. Initially, moisten a soft clean cloth, lens cleaning tissue or cotton swab to remove all foreign material from the glass bulb and/or platinum button. Then use a moistened cotton swab to carefully remove any material that may be blocking the reference electrode junction of the sensor. **CAUTION:** When using a cotton swab, be careful NOT to wedge the swab between the guard and the glass sensor. If necessary, remove cotton from the swab tip, so that the cotton can reach all parts of the sensor tip without stress. You can also use a pipe cleaner for this cleaning if more convenient.

If good pH and/or ORP response is not restored, perform the following additional procedure:

1. Soak the sensor for 10-15 minutes in clean water containing a few drops of commercial dishwashing liquid.
2. GENTLY clean the glass bulb and platinum button by rubbing with a cotton swab soaked in the cleaning solution.
3. Rinse the sensor in clean water, wipe with a cotton swab moistened with clean water, and then re-rinse with clean water.

If good pH and/or ORP response is still not restored, perform the following additional procedure:

1. Soak the sensor for 30-60 minutes in one molar (1 M) hydrochloric acid (HCl). This reagent can be purchased from most lab supply distributors. Be sure to follow the safety instructions included with the acid.



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2. Rinse the sensor in clean water, wipe with a cotton swab moistened with clean water (not DI water), and then re-rinse with clean water. To be certain that all traces of the acid are removed from the sensor crevices, soak the sensor in clean tap water for about an hour with occasional stirring.

If biological contamination of the reference junction is suspected or if good response is not restored by the above procedures, perform the following additional cleaning step:

**CAUTION:** Do not mix the acid from the previous step with the chlorine bleach in the following step. A toxic gaseous product can form from the reaction between the acid and the chlorine bleach. Be certain to copiously rinse the sink and drain system of acid after its disposal and before the disposal of chlorine bleach.

1. Soak the sensor for approximately 1 hour in a 1:1 dilution of commercially available chlorine bleach.
2. Rinse the sensor with clean water and then soak for at least 1 hour in clean tap water with occasional stirring to remove residual bleach from the junction. (If possible, soak the sensor for a period of time longer than 1 hour in order to be certain that all traces of chlorine bleach are removed.) Then re-rinse the sensor with clean water and retest.

Prior to reinstalling the sensor, dry the port and sensor connector with compressed air. If you suspect port contamination, follow the instructions in the Cleaning a Sensor Port section of this document before reinstalling the sensor.

If your pH sensor is still not calibrating after performing a sensor cleaning, contact your local YSI Representative or a YSI Authorized Service Center.

## Dissolved Oxygen

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The dissolved oxygen sensor should be calibrated every day the instrument is used. It is not necessary to calibrate in both % and mg/L or ppm. Calibrating in % will simultaneously calibrate mg/L and ppm and vice versa.

### CALIBRATION TIPS

1. The Pro Plus can be calibrated in air-saturated water, water-saturated air or against a Winkler Titration. You can perform a 1 or 2 point DO calibration. A 2 point calibration includes 1 point in a zero oxygen environment and the 2<sup>nd</sup> point at full saturation.
2. For both ease of use and accuracy, YSI recommends that you perform a 1 point calibration in water-saturated air.
3. Make sure that there is a good membrane with fresh electrolyte (O<sub>2</sub> probe solution) installed on the DO sensor. The membrane should be clean and free of wrinkles. There should not be any air bubbles present under the membrane. Membranes should be changed regularly and generally last 2-8 weeks depending on use and storage.
4. To perform a 1 point calibration in water-saturated air, place the sensor in a 100% humid environment. This can be accomplished several ways:
  - a. For the 60520 and 6052030 cables, moisten the sponge in the gray calibration sleeve with a *small* amount of clean water and place it over the sensor guard.
  - b. For the 6051020 and Quatro cables, place a small amount of water in the calibration/storage cup and place it over the sensors. When screwing the calibration cup onto the sensor bulkhead, only engage one or two threads. Do not screw the calibration cup completely onto the sensor bulkhead. The goal is to have air exchange between inside and outside the calibration cup.

The sponge and calibration sleeve/cup should be clean since bacterial growth may consume oxygen and interfere with the calibration. Be sure the sensor is in air, not water, and that there are not any water droplets on the membrane or temperature sensor.

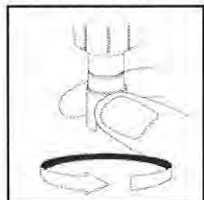
5. After entering the % calibration mode, wait approximately 5 to 15 minutes for the storage container to become completely saturated and, if using a polarographic sensor, to allow the sensor to stabilize.
6. Salinity affects the ability of water to hold oxygen and is used by the instrument to calculate DO mg/L (ppm). The Salinity value displayed near the top of the DO calibration screen is either the salinity correction value entered in the Sensor menu or the Salinity value as measured by the conductivity sensor in use. If you are using a conductivity sensor, ensure that it is calibrated and reading correctly in order to obtain accurate DO mg/L (ppm) measurements. If you are not using a conductivity sensor, the Salinity correction value should be the salinity of the water you will be testing. Highlight Salinity and press enter to modify this setting if necessary. The salinity of fresh water is typically 0-0.5 ppt and seawater is typically 35 ppt.
7. After accepting the calibration, navigate to the GLP menu and record the DO sensor's value (sensor current in uA). The acceptable sensor currents when calibration is performed at 25°C, in a 100% saturated air environment at 760 mmHg are:
  - 1.25 mil PE membrane (yellow membrane): Average 6.15 uA (min. 4.31 uA, max. 8.00 uA)
  - 2.0 mil PE membrane (blue membrane): Average 3.38 uA (min. 2.37 uA, max. 4.40 uA)
  - 1 mil Teflon membrane: Average 16.29 uA (min. 11.40 uA, max. 21.18 uA)
8. If you receive a warning message stating that the calibration is questionable, do not continue with the calibration. Instead, select 'No' and investigate what is causing the questionable results. If you accept a questionable calibration, your DO readings will be erroneous. Typical causes of a calibration error message include: incorrect sensor, membrane or port setup in the instrument, incorrect barometric pressure information, a bad membrane or a sensor that needs reconditioned.

## TROUBLESHOOTING TIPS

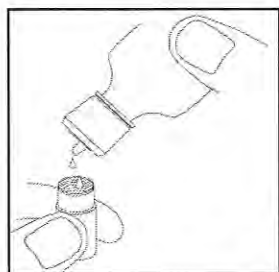
1. Ensure that the correct sensor type and membrane type are enabled in the Sensor Setup Menu. Galvanic sensors have a gray probe body and Polarographic sensors have a black probe body.
2. If using a 6051020 cable, ensure that the DO sensor is installed in port 2. If using a Quatro cable, ensure that the DO sensor is installed in the port labeled DO.
3. Ensure the Pro Plus barometer is reading accurately. The DO % Saturation calibration uses the instrument's barometric pressure reading for the DO % calibration. If the barometer is not reading accurately, the calibration will be erroneous. The barometer should be reading *true* barometric pressure. If you suspect the barometer reading is incorrect, calibrate the barometer and then recalibrate the DO sensor. Laboratory barometer readings are usually "true" (uncorrected) values of air pressure and can be used "as is" for barometer calibration. Weather service readings are usually not "true", i.e., they are corrected to sea level, and therefore cannot be used until they are "uncorrected". An approximate formula for this "uncorrection" is:  
True BP in mmHg = Corrected BP in mmHg – [2.5 \* (Local Altitude in ft. above sea level/100)]
4. Install a new membrane with fresh electrolyte onto the DO sensor. Ensure you are using the correct electrolyte solution. Polarographic sensors use electrolyte that is in a white labeled bottle (KCl/Na<sub>2</sub>SO<sub>4</sub>). Galvanic sensors use electrolyte that is in a blue labeled bottle (NaCl).
5. Recondition the DO sensor and then install a new membrane.
6. If you suspect port contamination, remove the sensor and follow the instructions in the Cleaning a Sensor Port section.
7. If you continue to have trouble calibrating the DO sensor, contact your local YSI Representative or a YSI Authorized Service Center.

## Membrane Cap Installation

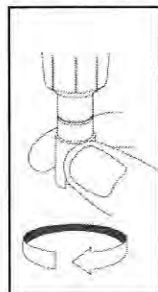
The DO membrane and electrolyte solution (O<sub>2</sub> solution) should be changed once every 2-8 weeks depending on use and storage. In addition, the membrane and electrolyte solution should be changed if (a) bubbles are visible under the membrane; (b) significant deposits of dried electrolyte are visible on the membrane; or (c) if the sensor shows unstable readings or other sensor-related symptoms. To install a new membrane cap follow these instructions:



1. Remove the sensor guard or cal cup to access the sensor tip.
2. Unscrew and remove any old membrane cap by holding the sensor when unscrewing the membrane cap. Discard the used membrane cap.
3. Thoroughly rinse the sensor tip with distilled or DI water.



4. Fill a new membrane cap with the appropriate electrolyte solution that has been prepared according to the directions on the bottle. Polarographic sensors use electrolyte that is in a white labeled bottle (KCl/Na<sub>2</sub>SO<sub>4</sub>). Galvanic sensors use electrolyte that is in a blue labeled bottle (NaCl). Be very careful not to touch the membrane surface during this process. Lightly tap the side of the membrane cap to release air bubbles that may be trapped.



5. Thread the membrane cap onto the sensor. It is normal for a small amount of electrolyte to overflow.

## Reconditioning the DO Sensor



### **Polarographic Sensors - Model # 605203**

Due to the chemical reaction taking place under the membrane, deposits will form on the gold cathode and silver anode. The gold cathode will begin to appear dull and the silver anode will turn dark in color. This discoloration is normal; however, it is recommended that you remove the deposits as needed. Perform the following cleaning procedures to remove the deposits if 1.) You have troubles calibrating the sensor or the DO readings are unstable; and 2.) Changing a membrane does not correct the problem.

#### **Silver Anode:**

After extended use, a layer of Silver Chloride (AgCl) builds up on the silver anode reducing the sensitivity of the sensor. The anode must be cleaned to remove this layer and restore proper performance. The cleaning can be chemical and/or mechanical:

Chemical cleaning: Remove the membrane cap and rinse the electrodes with deionized or distilled water. Soak the sensing electrode section of the sensor in a 14% ammonium hydroxide solution for 2 to 3 minutes or in a 3% ammonia solution overnight for 8-12 hours (most household ammonia cleaners are typically around 3%). Rinse heavily in cool tap water followed by a thorough rinsing with distilled or deionized water. The anode should then be thoroughly wiped with a wet paper towel to remove the residual layer from the anode. Trapping residual ammonia under the new membrane cap can quickly tarnish the electrode and/or give false readings.

Note: Chemical cleaning should be performed as infrequently as possible (1 or 2 times per year depending on use). First attempt a membrane change and recalibrate. If a new membrane does not resolve the problem, then proceed with cleaning.

After performing a chemical cleaning, perform a mechanical cleaning on both the anode and cathode.

Mechanical cleaning: In order to sand the silver anode along the shaft of the sensor, remove the membrane and hold the sensor in a vertical position. Wet 400 grit wet/dry sand paper with a small amount of clean water then gently wrap it around the sensor anode and twist it a few times to lightly sand the anode (the goal is to sand off any build-up without scratching or removing layers of the anode itself). Usually, 3 to 4 twists of the sanding disk are sufficient to remove deposits. However, in extreme cases, more sanding may be required to remove all of the deposits.

After completing the sanding procedure, repeatedly rinse the electrode with clean water and wipe with lens cleaning tissue to remove any grit left by the sanding disk. Thoroughly rinse the entire tip of the sensor with distilled or deionized water and install a new membrane.

**Gold Cathode:**

For correct sensor operation, the gold cathode must be textured properly. It can become tarnished or plated with silver after extended use. Never use chemicals or abrasives not recommended or supplied by YSI.

First dry the sensor tip completely with lens cleaning tissue. Wet 400 grit wet/dry sand paper with a small amount of clean water and place it face up in the palm of your hand. Next, with your free hand, hold the sensor in a vertical position, tip down. Place the sensor tip directly down on the sanding disk and twist it in a circular motion to sand the gold cathode. The goal is to sand off any build-up and to lightly scratch the cathode to provide a larger surface area for the electrolyte solution under the membrane. Usually, 3 to 4 twists of the sanding disk are sufficient to remove deposits and for the gold to appear to have a matte finish. Rinse thoroughly and wipe the gold cathode with a wet paper towel before putting on a new membrane cap.

Note: Be sure to: (1) Only use fine 400 grit wet/dry sand paper and (2) Sand as mentioned in the above procedures. Not adhering to either of these instructions can damage the electrodes. If this procedure is unsuccessful, as indicated by improper DO sensor performance, contact your local YSI Representative or a YSI Authorized Service Center.

**Galvanic Sensors – Model # 605202**

The Galvanic dissolved oxygen sensor is continuously reducing oxygen even when the Pro Plus is turned off. This factor allows the sensor to be used with no warm-up time as soon as the instrument is powered on. However, because the sensor is "on" all the time, some solid from the oxidation of the zinc anode will form in the electrolyte within 1-2 weeks of activation. The Galvanic electrolyte solution will appear milky white after use but this will not affect the accuracy of the sensor unless there is excessive build up which may result in jumpy readings. Otherwise, the color change is acceptable and normal as long as DO readings remain stable. ~~The rate of solid formation is dependent on the type of membrane installed. The formation of solids typically form more rapidly with the 5912 (black 1 mil Teflon), less rapid with 5913 (yellow 1.25 mil PE), and least rapid with 5914 (blue 2 mil PE).~~

When changing the membrane, rinse the anode and cathode with distilled or deionized water and wipe with a clean paper towel. If white deposits are evident on the anode after rinsing and wiping, remove the deposits by sanding the anode with 400 grit wet/dry sand paper following the "Mechanical Cleaning" instructions under the Polarographic Silver Anode maintenance section. If there are deposits on the cathode, sand the cathode with 400 grit wet/dry sand paper following the maintenance instructions listed for the Polarographic Gold Cathode.

Note: Do not perform the Polarographic chemical cleaning on a Galvanic sensor.

If this procedure is unsuccessful, as indicated by improper sensor performance, contact your local YSI Representative or a YSI Authorized Service Center.

## Installing and Uninstalling Sensors

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### GENERAL PRECAUTIONS

It is important that the entire sensor connector and cable connector be dry when installing, removing or replacing sensors. This will prevent water from entering the port. Once a sensor is removed, examine the connector inside the port. If any moisture is present, use compressed air to completely dry the connector or place directly in front of a steady flow of fresh air. If you suspect port contamination, follow the port cleaning procedures listed under Cleaning a Sensor Port.

Remove sensors upside down (facing the ground) to help prevent water from entering the port upon removal.

The instrument utilizes o-rings as seals to prevent water from entering the sensor ports. When the sensors are removed, the o-rings that provide the seal should be carefully inspected for contamination (e.g. debris, grit, etc.) and cleaned if necessary.

If no dirt or damage to the o-rings is evident, wipe the o-rings with a lint free cloth or lens cloth to remove the old o-ring grease. Then, lightly apply new o-ring grease (provided in the maintenance kit) to the o-rings without removing them from their groove. If there is any indication of damage, the o-ring should be replaced with an identical o-ring. At the time of o-ring replacement, the entire o-ring assembly should be cleaned.

Do not over-grease the o-rings. The purpose of the o-ring grease is to keep the o-ring in good condition. Excess grease may collect grit particles that can compromise the seal. Excess grease can also cause the waterproofing capabilities of the o-ring to diminish, potentially causing leaks. If excess grease is present, remove it using a lens cloth or lint-free cloth.

#### **To remove the o-rings:**

Use a small, flat-bladed screwdriver or similar blunt-tipped tool to remove the o-ring from its groove. Do not use a sharp object to remove the o-rings. Using a sharp object could damage the o-ring groove which would allow water to enter the port resulting in permanent damage to the port and sensor. Check the o-ring and the groove for any excess grease or contamination. If contamination is evident, clean the o-ring and nearby plastic parts with lens cleaning tissue or equivalent lint-free cloth. Alcohol can be used to clean the plastic parts, but use only water and mild detergent on the o-ring itself. Using alcohol on o-rings may cause a loss of elasticity and may promote cracking. Also, inspect the o-rings for nicks and imperfections.

Before re-installing the o-rings, make sure to use a clean workspace, clean hands, and avoid contact with anything that may leave fibers on the o-ring or grooves. Even a very small amount of contamination (hair, grit, etc.) may cause a leak.

#### **To re-install the o-rings:**

Place a small amount of o-ring grease between your thumb and index finger. Draw the o-ring through the grease while pressing the fingers together to place a very light covering of grease to the o-ring. Place the o-ring into its groove making sure that it does not twist or roll. Do not excessively stretch the o-ring during installation.

Use your grease-coated finger to once again lightly go over the mating surface of the o-ring.

Do not over-grease the o-rings. The excess grease may collect grit particles that can compromise the seal. Excess grease can also cause the waterproofing capabilities of the o-ring to diminish, potentially causing leaks. If excess grease is present, remove it using a lens cloth or lint-free cloth.

## **UNINSTALLING DO, PH, ORP, PH/ORP AND ISE SENSORS**

First, ensure that the entire sensor and cable bulkhead are clean and dry. Remove sensors upside down (facing the ground) to help prevent water from entering the port upon removal.

Simply unscrew the sensor from the cable by holding the sensor port end of the cable (bulkhead) in one hand and the sensor in the other hand. Twist the sensor counter-clockwise to unscrew the sensor from the port.

## **INSTALLING DO, PH, ORP, PH/ORP AND ISE SENSORS**

First, ensure both the sensor connector and sensor port on the cable are clean and dry. If any moisture is present, use compressed air to completely dry the connector or place directly in front of a steady flow of fresh air. If you suspect port contamination, follow the port cleaning procedures listed under Cleaning a Sensor Port.

To connect the sensor, grasp the sensor with one hand and the sensor port end of the cable (bulkhead) in the other. Push the sensor into the connector on the cable until it is properly seated and only one o-ring is visible. Failure to properly seat the sensor may result in damage. Twist the sensor clockwise to engage threads and finger tighten. Do not use a tool. This connection is waterproof. Please refer to the sensor installation sheet that is included with each sensor for detailed instructions.

## **UNINSTALLING A CONDUCTIVITY/TEMPERATURE SENSOR IN A QUATRO CABLE**

First, ensure that the entire sensor and cable bulkhead are clean and dry. Remove sensors upside down (facing the ground) to help prevent water from entering the port upon removal.

Remove the conductivity/temperature sensor using the installation tool to loosen the stainless steel retaining nut. Insert the tool into one of the holes in the stainless steel retaining nut. Next, use the installation tool to turn the stainless steel retaining nut counter-clockwise to loosen. Do not allow the sensor to be turned with the tool. Turning the sensor with the tool will likely damage the sensor connector. Once the stainless steel retaining nut has been completely loosened from the bulkhead, remove the sensor from the bulkhead by pulling the sensor straight out of the port.

## **INSTALLING A CONDUCTIVITY/TEMPERATURE SENSOR IN A QUATRO CABLE**

First, ensure both the sensor connector and sensor port on the cable are clean and dry. If any moisture is present, use compressed air to completely dry the connector or place directly in front of a steady flow of fresh air. If you suspect port contamination, follow the port cleaning procedures listed under Cleaning a Sensor Port.

1. Align the connectors of the sensor and the port. With connectors aligned, push the sensor in towards the bulkhead until you feel the sensor seat in its port. You will experience some resistance as you push the sensor inward, this is normal
2. Once you feel the sensor seat into the port, gently rotate the stainless steel sensor nut clockwise with your fingers, do not use the tool.
3. The nut must be screwed in by hand. If the nut is difficult to turn, STOP, as this may indicate cross threading. If you feel resistance or cross threading at any point, unscrew the nut and try again until you are able to screw the nut down completely without feeling any resistance. Damage to your cable/sensor may occur if you force the parts together.
4. Once completely installed, the nut will seat flat against the bulkhead. At this point, use the installation tool that was included with the sensor to turn the nut an additional  $\frac{1}{4}$  to  $\frac{1}{2}$  turn. Do not over tighten.
5. Please refer to the sensor installation sheet that is included with the conductivity/temperature sensor for detailed instructions.

## Cleaning a Sensor Port

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If you suspect port contamination, you can clean the port on the cable by filling the port with Isopropyl Alcohol for 30 seconds and then dumping it out. Next, allow the port to air dry completely or blow it out with compressed air. Installing a sensor into a port that is not completely dry is likely to cause erratic and erroneous readings.

If the connector is corroded, contact your local YSI Representative or a YSI Authorized Service Center.

## Verifying Sensor Accuracy and Calibration


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Sensor accuracy and calibration can be verified by immersing a sensor into calibration solution or YSI Confidence Solution®. Compare the readings on the Pro Plus display to the value of the solution. If the readings have drifted more than the accuracy specification of the sensor, perform a calibration before taking field measurements.

YSI Confidence Solution can be used to check the accuracy and calibration of the conductivity, pH and ORP sensors. However, to maintain the highest accuracy of the instrument, it should not be used to perform a calibration.

## Resetting a Sensor to Factory Default

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Occasionally, it may be necessary to reset the instrument to its factory calibration default values. To reset the calibration values, press the Cal key , highlight **Restore Default Cal** and press enter. Highlight the parameter you wish to reset to default and press enter. Next, you will be asked to confirm the operation. Highlight **Yes** and press enter to confirm.



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**Appendix G**  
**MPC Safety Procedure**  
**#12 GENERAL SAFETY RULES**

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Marathon Petroleum Company LP			
<b>Safety Procedure #12 GENERAL SAFETY RULES</b>	Document No. <b>1012</b>	Page <b>Page 1 of 31</b>	Revision No. <b>37</b>
	Original Issue Date 4/98	Revision Date <b>07-26-2019</b>	Next Revision Date <b>10-1-2020</b>
Document Custodian: <b>Safety</b>	Content Category: <b>Policy, Standard, Procedure or Guideline - General</b> Content Type: <b>Policy - Operating</b> Retention Period: <b>ACT + 10 years</b>		

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Appendix A – Building Fire Protection Policy Waiver

**Appendix B – Camera and Photography Procedure / Electronic Device Approval Form**

Appendix C – Electric and Instrument Shop Locked Vehicle Program

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Appendix I – PEP2 Device Evaluation Form

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**I. Purpose**

Define safe work practices not covered by specific Safety Procedures (SP). In addition to MPC Illinois Refining Division (IRD) employees, it is mandatory that contractors follow this SP and all other SP's.

**II. General Safety Rules**

**A. Smoking**

1. Smoking (both regular and electronic) is permitted inside designated areas only. Smoking (both regular and electronic) is prohibited in vehicles within the refinery fence and in all MPC vehicles at all times.

**B. Electronic Devices Policy**

1. There are three types of Electronic Devices covered under this policy.
  - i. Type I – MPC Owned or Approved Devices with an MPC Approved Rugged Case
  - ii. Type II – Approved Contractor Devices with a case that meets all minimum requirements listed below & has an MPC Refining Approval Sticker obtained from the Safety Supervisor.
  - iii. Type III – Personal Devices / Cell Phones

2. Contractor Device Approval Process

Contract Companies with a legitimate business purpose to use Contract-Company issued Electronic Devices in “Restricted” locations per the Electronic Devices Matrix must have those devices approved by a Department Manager by completing the approval form in Appendix H. Upon obtaining the approval form for business use on the device, the Contract Company must provide documentation that their device and/or device w/ case meets the minimum requirements listed below to the Safety Supervisor.

Contract Companies shall meet one of the following criteria:

- i. Electronic Devices clearly identified by or with factory labeling as “intrinsically safe”, “explosion proof”, or labeled as approved for use in hazardous locations rated as Class 1, Div. 1 or 2.
- ii. In order to use an Electronic Device in a hazardous area without a hot work permit, the Contract Company must establish a process consistent with the minimum requirements listed in Appendix I.

**NOTES:**

- Wrist Watches, Smart Watches, Fitness Trackers, and Medical Devices (e.g., hearing aids, etc.) are exempt from this policy.
- For PEP2 Medical Devices (e.g., insulin pump), user will wear a 4-Gas monitor in lieu of obtaining a hot work permit.

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		Electronic Devices Matrix			Use Notes
		Type I Devices	Type II Devices	Type III Devices	
<b>Restricted</b>	1	Allowed	Allowed	Not Allowed	A Electronic Device Approval Form provides "approved" status to a personal device. A business purpose is always required.
	2	Allowed	Allowed	Not Allowed	A Electronic Device Approval Form provides "approved" status to a personal device. A business purpose is always required.
	3	Allowed	Allowed	Allowed	Console Operators should not have their phones out on the console itself to avoid distraction. But may have it on their person or on the desk behind them. Personal use should be at a minimum. Excessive use is to be addressed by the direct supervisor. Others may use a device at the board if it is not a distraction to the board operator.
	4	Allowed	Allowed	Allowed	Non-working time is defined as during breaks or lunch
	5	Allowed	Allowed	Allowed	Cell phone use in vehicles is limited to passengers. The use of mobile phones while driving any vehicle is prohibited unless parked or using a hands-free device. A business purpose is always required.
	6	Allowed	Allowed	Allowed	Personal use should be at a minimum, excessive use is to be addressed by the direct supervisor.
<b>Approved Devices will NOT be allowed in Class 1, Division 1 Areas without a Hot Work Permit</b>					

3. Reference the Electronic Devices Matrix above for detailed guidance of Restricted Areas and where each type of device may be carried and/or used.
4. Cell Phone use in vehicles is limited to passengers, or when drivers are pulled over and parked at a complete stop or using a hands-free device. Cell Phones may not be used or on your person while operating a crane, man-lift or anything similar in nature
5. Even if powered off, Personal Devices/Cell Phones are unauthorized and not approved in Restricted Areas. An "Approved Device" refers to a cell phone that has been issued by MPC or "approved" by issuing the user an Electronic Device Approval Form found in Appendix H.

### C. Material Lifting

1. When lifting objects >55 lbs. you should utilize one of the following options:
  - i. Use two or more people to lift the load,
  - ii. Use mechanical means of lifting (fork lift, pallet jack, hand truck, etc.)

### III. Personal Protective Equipment

Personal protective equipment and safety devices must be used as required and must not be altered in any manner. The use of damaged or malfunctioning personal protective equipment is prohibited.

#### A. Safety Glasses with Approved Side Shields (ANSI - Z87.1)

1. ANSI approved safety glasses with side shields must be worn at all times within the refinery where work is being performed. This includes maintenance shop areas, the laboratory, and at

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designated work sites away from the refinery.

2. Safety glasses with side shields are **not** required to be worn in the following locations:

- a. **West of 2 ½ Street**
- b. Lunch/break rooms, control rooms, or plant offices
- c. Inside vehicles with enclosed cabs (windows closed)

Contact lenses may be worn in conjunction with safety glasses/side shields. Workers who wear contact lenses should inform the refinery nurse of their use. The nurse will issue hard hat stickers indicating contact use.

**B. Goggles and Face Shields**

- 1) Employees are required to have ANSI Z87.1 approved chemical splash goggles on their person (i.e., on their hard hat, in a pouch on their belt, etc.) when in process areas, the tank farm, or designated off site locations where the potential for flying debris or chemical exposure exists.

**NOTE:** Spoggles must not be used in place of goggles.

- 2) At a minimum, unless engineering controls are in place, the following requirements must be met:
  - a. A face shield OR goggles must be worn for the following jobs:
    - 1) Disconnecting hoses when potential for pressure exists.
  - b. Goggles must be worn for the following jobs or where there is risk of debris falling into the head/face area as a result of the work:
    - 1) Handling powdered, granulated or dusty materials and loose insulation. Note that if there is the need to use a dust mask or half mask particulate respirator, goggles still must also be used.
    - 2) Catching hydrocarbon samples.
    - 3) Using pressurized air, steam, etc. to clean equipment.
    - 4) Opening or transferring chemical totes via hoses.
    - 5) When performing any internal cleaning of dirt/debris in vessels, tanks, exchanger shells, furnaces, etc.
  - c. A face shield (over safety glasses) must be worn for the following jobs:
    - 1) A flying chip hazard exists (i.e, grinding, chipping such as concrete/refractory, cutting, buffing, blasting, etc.)
    - 2) While grinding or buffing vessels or equipment.
    - 3) When using a torch/wand to light burners on heaters or boilers.
    - 4) Operating an air powered nut gun/impact wrench.

**NOTE:** 3/8" and 1/2" battery powered impacts are excluded when used with impact sockets.

- 6) When handling/working with hot products 140° F (molten sulfur, hot resid, hot condensate/boiler feedwater, etc.)
- 7) Operating a string trimmer during lawn maintenance.

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8) When looking into fired heaters/boilers

d. A face shield AND goggles must be worn for the following jobs:

- 1) Connecting/disconnecting lines or hoses in acid or caustic service.
- 2) When catching samples in acid or caustic service.
- 3) Cleaning, draining or repairing equipment which has been in acid or caustic service and not neutralized.
- 4) Loading or unloading of acids or caustics.
- 5) Initial line breaking or opening of equipment when potential for pressure exists.
- 6) Open sampling of liquids/products above 140 degrees F (non-engineering sample systems).

**NOTE:**

- 1) Goggles and/or face shields are not required when using a full-face respirator such as with fresh air equipment.

C. Safety Toe Shoes (ASTM F2413)

ASTM approved safety toe shoes with at least a ¼” defined heel must be worn at all times within the refinery property and at designated work sites away from the refinery when work is being performed.

ASTM approved shoes are not required to be worn in the following locations:

1. Lunch/break rooms, control rooms, plant offices
  2. Inside vehicles
  3. Employees reporting to work or leaving work provided they go directly to their work area.
  4. Walking directly to or from personal vehicles or offices outside process unit battery limits.
  5. Truck drivers and vendors making deliveries or pickups of supplies.
6. Laboratory – shoes must be made of leather, rubber, or other non-absorbing material.

**NOTES:**

- Metatarsal guards must be worn on ASTM approved shoes when using a jackhammer or when hydroblasting.
- Open-toed shoes, sandals, & high-heeled shoes are not permitted inside the refinery.

D. Head Protection (ANSI Z89.1 Type 1 Class “E”)

All employees are required to wear an ANSI Z89.1 Type 1 Class “E” approved hard hat when in process areas, tank farm, designated off site locations where work is being performed, or new construction areas.

1. Hard hats must be changed at a minimum of every five years from the born-on date or when damaged or showing visible signs of wear (i.e. cracks, disfigurement, UV Damage etc.)
2. Hard hat suspensions must be changed at least annually
3. Hair length longer than the shoulders must be kept under a hardhat when working around rotating equipment.

E. Flame Resistant (FR) Protective Clothing



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These procedures must be adhered to in order to provide adequate protection for workers in areas where there are recognized fire hazards and a reasonable probability that FR could mitigate burn injuries.

- 1) All FR clothing base garments (shirt/pant combo and/or coveralls) shall either be inherently FR material (e.g., Nomex, PBI) or FR treated cotton and cotton blends that are certified by an independent testing agency meeting NFPA 2112.
- 2) Seasonal accessories (e.g., UV face masks, cold weather beanies, or hard hat liners) shall also be meet NFPA 2112. (**RSP Compliance Date - January 1, 2020**)
- 3) Garments worn underneath base layers for warmth/cooling shall be made of natural fibers such as cotton, wool, or silk. This requirement does **not** include underwear.

**IMPORTANT:** Base layers made from synthetic materials such as polyester (e.g., Under Armor) are **Prohibited**.

- 3) FR shirts (not including outer FR garments (e.g., coats and sweatshirts with or without hoods, etc.) shall be tucked in, buttoned up, and sleeves rolled down when in FR required areas to comply with **NFPA 2113**.
- 4) Outer FR garments (e.g., coats, bibs, and sweatshirts with or without hoods, etc.) shall be made of FR fabric and adhere to **NFPA 2112** requirements that are certified by an independent testing agency.
- 5) **NFPA 2112** daily FR work wear garments shall be worn at all times under all outer FR garments.
- 6) Hole watch/Fire watch vests shall comply with **ASTM D6413** Flame Resistant requirements.

**FR Rain Wear:** (**RSP Compliance Date - January 1, 2020**)

- 7) All rain wear shall comply with **ASTM D6413** Flame Resistant requirements, and shall be tested and comply in accordance with:
  - a. **ASTM F2733** for flash fire, and
  - b. **ASTM F1891** when the risk potential of an arc flash hazard exists.

**FR Disposable Coveralls:**

- 8) Disposable coveralls shall be made of FR fabric and are not required to meet NFPA 2112 requirements.
- 9) Disposable coveralls shall comply with ASTM D6413.
- 10) Disposable coveralls shall comply with NFPA 2113 as it pertains to the care and maintenance during use.

**NOTE:** Any garments soiled with hydrocarbons or visibly tattered during work activities must be removed from service and replaced.

Each employee shall be responsible for the inspection and integrity of fire-resistant garments issued to them. Employees shall routinely inspect the garments for rips, tears, holes, discoloration, function of buttons, zippers, and fabric thinning due to age and repeated washings. Damaged clothing should be repaired or replaced.

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FR shall be worn by all personnel in the refinery with the following exceptions:

- a. Employees will be allowed entry into the refinery while wearing dresses, sleeveless shirts, & short pants, west of 2<sup>nd</sup> Street and including the E&I Shop, Main Warehouse, or while riding in an enclosed vehicle to Complex / PDU / Lab break rooms.
- b. Employees reporting to work and leaving work, provided they go directly to their work area.
- c. In Control Rooms and offices that are outside process unit battery limits.
- d. Inside the Warehouses, E & I Shop, Machine Shop, Welding Shop, the Garage and Firehouses provided that no threat of flash fire exist.
- e. While in the offices, main hallways and lunch/break rooms in the Laboratory.
- f. In new construction areas that are not in an operating unit.
- g. On refinery roadways.

F. Hand Protection

Gloves must be worn for jobs that have the potential for hand injury. Each person when in process areas, the tank farm, or designated off site locations where the potential for hand injury exists who is required to wear fire resistant clothing shall at least have general duty work gloves conforming to ANSI/ISEA 105 Level 3 at least in the palm, fingers and thumb of the glove for general operations and maintenance work. These gloves are not a substitute for protective chemical gloves, as required in the site-specific PPE requirements and minimum requirements listed in Appendix F.

For tasks with the potential of impact hazards, gloves with impact protection to the back of the hand and full length of the fingers are to be worn. (e.g., work with hammers, picking up blinds/valves, hand wrenching flange bolts, impact gun tasks, tasks where hands and fingers can be pinched between the tool and a fixed object or material)

G. Hearing Protection

Hearing protection is required to be worn inside the operating boundary (perimeter) of all process units, including during shutdown/turnaround periods. High noise areas in the plant may be designated by a yellow stripe and/or signs stating "Caution - Ear Protection Must Be Worn In This Area". High noise areas are also encountered around operating equipment such as vacuum trucks, compressors and operating pumps in the tank farm. Hearing protection must be worn regardless of the time spent in these areas.

H. Life Jackets

U.S. Coast Guard-approved life jackets must be worn at all times whenever there is a danger of falling into a body of water and 100% fall protection cannot be maintained. This includes barges, floats (without hand rails), rowboats, motorboats, or any other equipment in or over water.

When wearing a life jacket or work vest it should be adjusted and the top and bottom buckles fastened.

Prior to and after each use, the life jacket or work vest must be inspected for defects which would alter their strength or buoyancy. Defective units must not be used.

I. Hydro Blasting

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When performing hydroblasting operations, the following personal protective equipment must be worn in addition to normally required PPE:

1. Face shield and safety glasses must be worn for eye/face protection.
2. A rain suit must be worn for skin protection from splashed liquids.
3. Gloves must be chosen based on the material to be encountered.
4. Chemical protective boots shall be worn while blasting. The boots must meet ANSI Z41 requirements and provide metatarsal protection.

The following Hydro Blasting requirements must be strictly adhered to:

1. When using a flexible lance, the operator of the lance must also operate the pressure control peddle.
2. An anti-withdraw device, anti-whip checks, and an appropriate stinger to prevent the lance from turning around must be used for all flexible lance operations.
3. The hose and nozzle size for flexible lance operations must be appropriate for the job.
4. Hydro blasting must be conducted by trained personnel in accordance with hydro blasting procedures.

#### J. Abrasive Blasting

When performing abrasive blasting operations, the following personal protective equipment must be worn in addition to normally required PPE:

- a. Kevlar sleeves must be worn to protect the arms.
- b. Gauntlet Cuff Canvas Gloves or Leather Gloves with ANSI Cut Level 3 must be worn.
- c. Supplied air blast hoods must be worn during all abrasive blasting activities.
- d. Personnel working around the blasting area that may be exposed to general dust must wear a half mask respirator with P100 cartridges.
- e. Personal CO monitors must be worn to detect the buildup or presence of Carbon Monoxide.
- f. FR Tyvek is required in process areas.

The following Abrasive Blasting requirements must be strictly adhered to

- g. All hoses must have whip checks and all nozzles must be equipped with a dead man's switch.
- h. Establish a means of communication between the blaster & pot man for confined space work.
- i. Abrasive blasting must be conducted by trained personnel in accordance with blasting procedures.

#### IV. Hand Tools

- A. Check tools before use to be certain they function properly and are suitable for the job.

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1. Hand-held power tools must be equipped with a constant-pressure switch or control that shuts off the power when pressure is released. Hand tools with a “lock-on” control switch are not permitted to be used.
- B. **When operating a pneumatic or hydraulic torqueing/de torqueing tool, consideration should be given to hand placement so that the employee is not putting themselves in the line of fire or placing their hands in a pinch point.**
- C. When job tasks require the use of electrical tools/equipment and/or electrical extension cords, a self testing GFCI must be utilized.
- D. All Pocket Knives used on IRD Property are required to have a locking blade.

**V. Safety in Moving Through the Refinery**

A. Barricades and Road Closings

A “barricade tag” must be placed on all blockades (barricades, flagging, netting, tape, etc.) indicating the reason, the nature of the hazard, and the name of the person installing the tag except during plant emergencies.

Restricted areas must be adequately barricaded, such as utilizing netting, barricades, warning tape, and/or scaffolding.

**Driving around barricades, flagging, cones, etc., used to block a road is prohibited.**

1. Barricades will be used around equipment or objects on or near the sides of roadways to make personnel aware of possible hazards in that area.
2. Holes or restricted areas not in or near roadways must be adequately barricaded, such as through netting or barricades.
3. Temporary pipe crossings in roadways will have a barricade on each side of the road to make personnel aware of the hazard.
4. Anytime barricades are required, including emergency situations, Security must be contacted to inform them what needs barricaded and the specific restrictions.
5. In order to maintain minimum traffic access, the following minimum considerations will apply:
  - Of the two major North/South roads (**2 ½ St.**, on the West side of the NHT/Platformer, HF Alky, Sat Gas and **3<sup>rd</sup> St.** on the East of the NHT/Platformer, HF Alky, Sat Gas) only one may be closed at any given time.
  - Of the three major East/West roads: (“**H**” **St.**, on the South side of Sat Gas, Sour Water Stripper, CX-1 BRM; “**J**” **St.**, on the North side of bullets/spheres; and “**K**” **St.**, on the South perimeter fence line); only two may be closed at any given time.
  - Of the two roads on the North and South side of the Main Warehouse, only one may be closed at any given time.

**Any deviation from the minimum requirements listed above must be reviewed by the General**

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**Maintenance Supervisor, Operations Shift Foreman, & Safety Supervisor with final approval from the Safety Supervisor.**

6. If an individual blocks a road or area themselves, Security must be notified of what type of barricades have been put in place and the specific restrictions. (Be sure to follow instructions for placement depending on the degree of restrictions.)
7. When a roadway and areas are open, notify Security to pick up the barricades.
8. Security notifies the appropriate Ops/PDU Shift-Foreman, Laboratory and the Safety Department of all road closings.

**B. Driving Through Fog**

The speed limit is 5 mph when driving through fog. When possible, alternate roadways must be taken when fog makes visibility very limited.

**C. Spotter Usage Requirements for Vehicles**

Prior to entering process units, insure provisions (spotters, barricades, etc.) are in place to prevent contact of the vehicle with process equipment. Consideration shall be given if a spotter will be required on roads not normally open to traffic, construction sites, or in heavily congested areas.

**VI. Forklift Safety**

**A. General Requirements**

The following procedure has been developed to identify basic forklift safety requirements. *Forklifts are also commonly referred to as fork trucks.*

1. Forklifts must bear a label or some other identifying mark indicating approval by a testing laboratory.
2. Modifications and additions which affect capacity and safe operation must not be performed without manufacturer written approval.
3. Only properly trained personnel are permitted to operate forklifts.
4. Telescoping Forklifts must be equipped with a low boom configuration for optimum vision.
5. Personnel must not stand or pass under the elevated portion of the forklift, whether loaded or empty.
6. Forklifts operating near the edge of ditches, embankments, ramps, docks, etc., must maintain a minimum of one foot clearance on both sides.
7. Fire aisles, stairway accesses and fire equipment must be kept clear.
8. Only stable and safely arranged loads shall be handled, and the loads must be within the rated capacity of the forklift.

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9. If at any time a forklift is found to be in need of repair or is defective, which creates an unsafe condition, it must be taken out of service until it has been restored to safe operating condition.
10. Fuel tanks must not be filled when the engine is running. Spilled fuel or oil must be washed away and the filler cap replaced before starting the engine.
11. Do not operate forklifts without proper authorization inside process unit battery limits, tank dikes or other areas where flammable vapors may be present.
12. Telescoping Forklifts are prohibited from operating inside refinery warehouses.
13. Forklifts must be equipped with adequate lighting.

**B. Traveling**

1. Drivers are required to look in the direction of, and keep a clear view of the path of travel.
2. Drivers must slow down and sound the horn at cross aisles and other locations where vision is obstructed. If the driver does not have an adequate field of view, the driver is required to travel with the load trailing.
3. MPC owned, leased, or rented Telescoping Forklifts are prohibited from transporting material on public roadways (city streets) except in emergency situations as authorized by the Emergency Control Center.
4. On all grades the load and forks must be tilted back if applicable, and raised only as far as necessary to clear the road surface.
5. Special caution must be taken when moving or working on inclines/declines, wet or otherwise slippery surfaces.
6. Forklifts must not be used to transport excessively long and/or unstable loads of lumber, pipe, etc.
7. All traffic regulations must be observed, including authorized plant speed limits.

**C. Material Lifts with a Forklift**

1. Piping, or any other material, Shall not be picked up with a sling, shackles, rings or chains that are rigged from the forks of a fork lift without the manufacturers written approval.
2. It is acceptable to lift piping, or any other material, with an engineered device approved by the forklift manufacturer.
3. Piping must not be transported by positioning the fork into the end of the pipe.
4. The following applies to material that is being transported from a lay-down area to the work site with a forklift.

- a. All material must be stable and/or strapped to the forklift.

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- b. It is acceptable to transport pre-fabricated pipe spools with a forklift as long as the material is stable and/or strapped to the forklift.
  - c. The maximum allowable length of material to be transported by a forklift is 24 feet. Material lengths longer than 24 feet shall be transported by means other than a forklift.
  - d. A spotter shall be provided if the operator does not have an adequate field of view and/or when transporting any load longer than 15 feet. The spotter may be on foot or use a motorized vehicle as appropriate.
5. The following applies when piping or structural steel material is being unloaded from a truck in a designated lay-down area with a forklift.
- a. There are no length restrictions on piping or other materials being unloaded with a forklift.
  - b. Piping or other material does not need to be strapped to the forklift.

**VII. Building Fire Protection**

- A. In order to reduce the likelihood of a fire in building and trailer offices the items listed below shall be followed:
- 1. Approved appliances (e.g., industrial coffee pots and approved refrigerators) will only be allowed in designated areas (see SP #12 Appendix D).
  - 2. Appliances that say “household use” or “for household use only” shall not be used.
  - 3. Individual offices and storage areas shall not have heat producing appliances such as: coffee pots, space heaters, electrical hot plates for cups, microwaves, refrigerators, temporary lighting, potpourri pots and toasters. Designated kitchen areas and break rooms are allowed heat producing appliances (see SP #12 Appendix D).
  - 4. Only extension cords provided by the MPC Electricians shall be used.
  - 5. Do not run power cords under carpet or rugs.
  - 6. Do not store combustible materials immediately adjacent to electrical equipment.
  - 7. Only approved electrical surge protectors in good condition can be used. Surge protectors should be UL Rated and be labeled "Transient Voltage Surge Protector". Approved electrical surge protectors can be obtained from the Office Services Supervisor and should be labeled with an “Electric Department” tag. UL rated surge protectors are required
  - 8. Report suspicious hot odors to your supervisor/manager. Complete a detailed search until the source is found. If the source is not found, the supervisor/manager must report the suspicious hot odor to the Safety Supervisor or Refinery Fire Chief.

**NOTE:** Exceptions to rules 2 through 7 require a waiver signed by an Electrical Competent Person, listed in SP #24 and the effected Department Manager. (See SP #12 Appendix A for waiver)

- B. At the discretion of Department Managers fire proof file cabinets will be used to protect critical documents.
- C. At the discretion of Department Managers the “Cozy Toes” foot warmer by TriLite Inc., may be used by employees.

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**VIII. Compressed Gas Cylinders**

- A. All compressed gas cylinders must be stored upright and fastened securely (chained or roped off) to prevent falling.
- B. Protective caps must be kept on when cylinders are not in use. Cylinders must never be picked up or transported by hooking a line to the cap or cylinder.
- C. Always use, transport and store full or empty cylinders in a vertical - not horizontal - position with the valve end up. Use specifically designed holders for moving cylinders by hoist, crane or truck. Do not use slings.
- D. Oxygen cylinders must be stored at least 20 feet from all flammable gas cylinders (acetylene, hydrogen, etc.) or separated from them by a firewall at least 5 feet high with a one half hour fire resistance rating.
- E. All cylinders must be properly identified with labeling or stenciling.
- F. When cylinders are left unattended with hose and torch still connected, cylinder valves must be closed to prevent accidental gas release.
- G. Keep oil away from cylinder valves.
- H. Do not store cylinders next to heat sources.
- I. When a cylinder is empty, close the valve and mark the cylinder "EMPTY" or "MT".
- J. Should a cylinder safety valve relieve and fire start, cool the cylinder. Do not attempt to extinguish the fire.

**IX. Area Color Codes Used in the Refinery**

- A. Blue - General Cooling / Utility Water and Unit Boundary Limits
- B. Yellow Stripe on Concrete/Yellow Signs - High Noise Area
  - 1. Can be designated by a yellow stripe on the pavement and/or signs stating, "Caution - Hearing Protection Required".
- C. Neon Green or Neon Yellow - Safety Showers and Eyewash Stations
- D. Orange with Yellow Stripe - Hydrofluoric Acid Areas
  - 1. Designated by an orange line with a yellow stripe in the middle.
  - 2. Personal protective equipment must be worn in these areas as mandated by Department Policy and/or your immediate supervisor.
- E. Yellow Flanges - All flanges in HF Acid and "trace" HF Acid service. The yellow acid detecting paint will turn red if exposed to HF Acid.
- F. Red and Yellow Stripe - Caustic Areas
  - 1. Personal protective equipment must be worn in these areas as mandated by Department Policy and/or your immediate Supervisor.



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G. Red Stripe - Sulfuric Acid Areas

1. Personal protective equipment must be worn in these areas as mandated by Department Policy and/or your immediate supervisor.

H. Red - Fire Fighting and Fire Protection Equipment

1. Fire extinguishers, hydrants, monitor nozzles, fixed systems, foam cabinets, etc.

I. Green - Nitrogen Systems

**X. Refinery Area Ownership/Responsibility Guidelines**

A. Permanent Lay-Down Area

1. Several areas in the refinery have been designated as permanent lay-down areas for maintenance groups, maintenance contractors, or project groups.
2. Designated lay-down areas shall have their boundaries marked in the field. Generally posts will be adequate and are preferred for these areas. However, when better control of the area or materials is needed, fencing may be used. When only posts are used for marking the area, they should be at each corner of the lay-down area and not more that 30 feet apart. Storage of equipment and materials must be kept within these boundaries.
3. A sign must be posted at the lay-down area indicating the group responsible for the area.

B. Temporary Lay-Down Area

1. Temporary lay-down areas are often necessary to store incoming materials for project work. When there is a need to establish a lay-down area, permission must be requested from the owning supervisor of the refinery area wanted for the lay-down area.
2. When requesting to use an area in the refinery for a lay-down area, the following information must be provided to the owning supervisor of the area.
  - Name of the project requesting the area
  - Marked up plot plan indicating area requested (joint site visit may be necessary)
  - Size of area being requested
  - Time frame the lay-down area will be needed
  - Contents that will be stored in the area
  - MPC representative that will take responsibility for the area while in use as a lay-down area
3. Once an area is approved for a lay-down area, temporary responsibility for upkeep/maintenance (including bomb searches) of the area will be with the project using the area.
4. Boundaries of the lay-down area must be established and marked in the field by posts or fencing to control use of the area. Use of areas outside of these boundaries is not allowed.
5. A sign shall be posted at the lay-down area indicating the project using the area.

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6. Prior to a lay-down area being returned to the original owner, the area must be restored to its original condition or a condition agreed upon by the original owner, including removal of all surplus materials.

**NOTE:** When temporary storage of materials or equipment is needed for two weeks or less, the owning department may waive some or all of these requirements. The person requesting the lay-down area must still receive approval from the owning department prior to use.

C. Temporary Fabrication Area

1. Temporary field fabrication areas without enclosed fabrication structures are often necessary for project or maintenance work. When there is a need to establish a field fabrication area, permission must be requested from the owning supervisor of the refinery area wanted for the fabrication area. If hot work is to be performed at the location, approval is also required from the Safety Supervisor.
2. When requesting to use an area in the refinery for a fabrication area without enclosed fabrication structures, the following information must be provided to the owning supervisor of the area. Approval is also needed from the Safety Supervisor if hot work is involved.
  - Name of the project requesting the area
  - Marked up plot plan indicating area requested (joint site visit required when hot work is to be performed)
  - Size of area being requested
  - Time frame the area will be needed
  - Scope of work intended for the area (e.g. storage of materials, hot work, etc.)
  - MPC representative that will take responsibility for the area while in use as a fabrication area
3. Boundaries of the fabrication area must be established and marked. Use of areas outside of the boundaries is not allowed.
4. A sign should be posted at the area indicating the project using the area.
5. Prior to a fabrication area being returned to the original owner, the area must be restored to its original condition or a condition agreed upon by the original owner, including removal of all surplus materials.

E. Pipe Rack Ownership

1. Definitions:
  - a. Ownership: Operate, maintain, permit, control access to a particular piece of equipment or area.
  - b. Battery Limits: The area of the pipe rack at the edge of a process unit where blinds are installed to isolate a unit during turnarounds or other maintenance functions. It often coincides with the blue lined area of the unit. This will denote the location where ownership of the line transfers from Area 4/5 to the units' operating area. The battery limits block valves are owned by the unit.

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- c. Process Areas: The Areas that contain the refinery process units and include Area 1, 2, 3 & 4.
- d. Utilities: For the purpose of assigning ownership of the utilities the following are considered utilities owned and maintained by Area 4 when located outside of a process unit:
  - Steam – High, medium, and low-pressure headers
  - Condensate
  - Instrument Air
  - Plant Air
  - Nitrogen
  - Boiler Feed Water
  - Raw water supplied to the plant from Palestine wells and pit
  - Electrical lines
  - City Water
  - Cooling Water
  - Utility water system
  - Sanitary sewer system
- e. Other refinery wide systems considered owned by Area 5 when located outside of a process unit.
  - Firewater system
  - Steam tracing system on piping outside of units
- f. Instrument lines are owned and maintained by the Area who monitors the instrument reading. L&J wiring is owned by Area 5.
- g. Area 1 is the owner of the TDC highways and the associated cable trays.

2. Pipe Rack Ownership Guidelines

The pipe racks throughout the refinery are assigned ownership as follows:

- Within the process units – all ownership is assigned to the respective unit's Area and they are responsible for all operation, maintenance, and permitting
- Outside the process unit battery limits – the pipe rack structure and piping within the rack is assigned to Area 5 with the exception of the utility piping located in the pipe rack. The utility piping will be owned and maintained by Area 4. All electrical lines and poles are also assigned to Area 4 for ownership and maintenance.
- Pipe racks that just contain utility piping will be assigned to Area 4 for ownership and maintenance, including the pipe rack structure.
- Housekeeping issues beneath the pipe racks, and to the middle of the road, are the responsibility of the adjoining property owner, not Area 5.
- Buildings located under pipe racks are the responsibility of the Area using the building and are not considered part of the pipe rack. Any pipe rack structural members that are integral to the building will be maintained by Area 5 and any required work will be coordinated with the building's owner.

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3. Underground Piping

- a. Underground piping outside of the process unit battery limits is owned and maintained by Area 5 with the exception of any utility piping as defined above.
- b. Sewer piping, including sanitary sewers, within the process units is maintained by the respective process Area.
- c. Firewater and Utility water piping and equipment ownership is determined by the Fixed Safety Equipment list. Area 5 owns and operates the firewater valves and would be responsible for isolating a particular monitor or section of firewater or utility water piping. The Process Areas are responsible for maintaining the equipment and associated piping listed on their Fixed Safety Equipment lists including the underground piping up to the valve located at the main line tie in point. All work orders for firewater and utility water repairs are charged to a Unit 24 work order.
- d. The transfer of ownership between the Process Area and Area 5 occurs at the junction between the branch and the main header line.

4. Railroad Ownership Guidelines

The railroad track is owned and operated by the Area that uses the track and as indicated on the [Refinery Area Ownership/Responsibility Plot Plan Drawing E-053526](#). ~~Area 1 completes any rail repairs identified.~~

**XI. Guidelines for Setup of Temporary Equipment near Fixed Firefighting Equipment**

- A. If access to, or water discharge from any hydrant and or hydrant/monitor combination is blocked by mobile equipment, a temporary structure, or construction activities, etc. for any period of time, the Fire Chief and/or Area Safety Representative must be contacted to determine if an alternate means of protection and/or mitigation resource is warranted.

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## REVISION HISTORY

Revision Number	Description of Change	Written by	Approved by	Revision Date	Effective Date
0	Original Issuance	SOP Review Team	Refinery Mgt. Team	4/98	4/98*
1	Changes to: II.B; V.E; Appendix E	SOP Review Team	Refinery Mgt. Team	7/06	7/06
2	Changes to: II, C Lightning	SP Review Team	Refinery Mgt Team	7/07	7/07
3	Change to: IV. Hand Tools	SP Review Team	Refinery Mgt. Team	8/07	8/07
4	Changes to: II.D; III.I; V.A; VI; Appendix C	SP Review Team	Refinery Mgt. Team	7/08	7/08
5	Addition of Section XVII	SP Review Team	Refinery Mgt. Team	12/08	12/08
6	Change to: V.A.1.a. & b.	SP Review Team	Refinery Mgt. Team	3/09	3/09
7	Updated Appendix D	SP Review Team	Refinery Mgt. Team	7/09	7/09
8	Updated XV. & App. C	SP Review Team	Refinery Mgt. Team	12/09	1/10
9	Updated IIB; IIIB, C, E; VA; VIH; Appendix A, C, D, E, F	SP Review Team	Refinery Mgt. Team	10/10	10/10
10	Updated: III.C. & D.; V.C.; Appendix E & F	SP Review Team	Refinery Mgt. Team	5/11	5/11
11	Updated III B, C, D, & G	SP Review Team	Refinery Mgt. Team	10/11	10/11
12	Changes to: V.C.	SP Review Team	Refinery Leadership	2/12	2/12
13	Change to XV	Safety Supervisor	Refinery Leadership	3/12	3/12
14	Changes to III B, XV, Added App G	Ron Clouse & SP Rev. Tm	Refinery Leadership	5/12	5/12
15	Changes to II, XII & added 2 <sup>nd</sup> link	Safety Supervisor	Refinery Leadership	9/12	9/12
16	Appendix E Corrected	Ron Clouse – PSM	Safety Supervisor	2/13	2/13
17	Corrections to Section II A & C	Safety Professional	Safety Supervisor	7/13	7/13
18	Changes to Section II. B & E	Safety Supervisor	Refinery Leadership	8/13	8/13
19	Changes to Sections II.G., XIV & App. F	Safety Professional/SP Review Team	Refinery Leadership	10/13	10/13
20	Clarification to App C	Safety Professional	Safety Supervisor	8/14	8/14
21	Three Year Review – Changes to Section III. B.	SP Review Team	Refinery Leadership	10/14	10/14
22	Changes to Section III.	Safety Professional	Refinery Leadership	5/15	5/15
23	Changes to Section XIV	Safety Professional	Safety Supervisor	7/15	7/15
24	Changes to Section XIV	Safety Professional	Safety Supervisor	8/15	8/15
25	Change to Section II. C	Safety Department	Refinery Leadership	11/15	11/15
26	Change to Section II, Section III, Appendix C, & Added Appendix H	Safety Department	Refinery Leadership	2/16	3/16
27	Added Section IV. "Hydraulic Equipment"	Safety Supervisor & SP Review Team	Division Staff	3/16	4/16
28	Changes to Section XVI E.	PDU Supervisor & Fire Chief	ES&S Manager	6/16	6/16

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29	Added Section II, E "Army Pipe", and XIV, B "Machine Guarding Reference"	J.D. Trimble	Division Staff	9/16	12/16
30	Addition to Section V., Added Section III. L. & Appendix I	Safety Professional & SP Review Team	Division Staff	11/16	1/17
31	Addition to Section II. & Added Section XVIII.	Safety Professional & SP Review Team	Division Staff	3/17	6/17
32	Changes to Section II. B. & Added Appendix K	Safety Department	Division Staff	9/17	9/17
33	Replaced KMS reference with Electronic Management System	Safety Technician	Safety Supervisor	6/18	6/18
34	3 YR Review – Procedure Rewrite	Safety Professional & SP Review Team	Division Staff	10/17	9/18
35	Changes to Section IV.	Safety Professional	Safety Supervisor	10/18	10/18
36	Changes to Section III per RSP-1716-000 implementation	Safety Professional	Safety Supervisor	12/18	1/18
37	Changes to Sections II.C; III.A; IV; V.C; XI. Appendix B	Safety Professional & SP Review Team	Division Staff	7/19	7/19

\* **NOTE:** The Revision History Table was implemented in 2006.

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## Appendix A

MARATHON PETROLEUM COMPANY LP  
Illinois Refining Division

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

### **WAIVER OF BUILDING FIRE PROTECTION POLICY**

A waiver of the Marathon Petroleum Company LP Building Fire Protection Policy is being issued for

\_\_\_\_\_, (employee name, group, or location) for the

purposes of performing the following at the specified location within the refinery:

\_\_\_\_\_  
\_\_\_\_\_

This waiver is valid for the following dates: \_\_\_\_\_

\_\_\_\_\_  
Electrical Competent Person  
Illinois Refining Division  
(Per SOP #12 – Building Fire Protection)

\_\_\_\_\_  
Department Manager  
Illinois Refining Division  
(Per SP #12 – Building Fire Protection)

#### **NOTES:**

- **If this waiver is required to address a medical condition, please submit this form directly to the Refinery Nurse. You will be notified if additional information is required. If it is not for a medical condition, please complete the following steps:**
  - The original copy of this waiver must be forwarded to the Safety Department.**
  - A copy of this waiver must be kept at the location for which it was issued.**
  - The waiver can only be written up to a 12 month period.**

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### **Appendix B - Camera and Photography Procedure**

Any photographic equipment, including still cameras, video cameras, cell phones when used as a camera, and any other device capable of capturing and storing an image will be considered a camera for this procedure. Safe work practices must be followed when using a non-intrinsically safe camera, cell phone, or tablet without an intrinsically safe case within unit battery limits and tank dikes. Intrinsically safe cameras, cell phones, and tablets with intrinsically safe cases installed per manufacturer's instructions are exempt; therefore work clearance permits, atmospheric monitoring, etc. are not required for these cameras, phones, or tablets within the unit battery limits, tank dikes and any other areas of the refinery property.

#### **Marathon Employees**

1. Marathon employees are not required to obtain written authorization or a camera pass.
2. All photos and videos remain Company property and cannot be distributed outside the Company without the Department Manager's approval, or his/her designee. Photos to be used outside the Company for publications, public presentations, etc. must be provided to the local Human Resources Department who will obtain Corporate Public Affairs approval for their use.

#### **Contract Employees**

1. Must obtain an **Electronic Device Approval Form (Appendix H)** from his/her Marathon contact. **Electronic Device Approval Sticker will not be required for a camera.** The form must be filled out and signed by the Department Manager or his/her designee. **An Electronic Device Approval Form** will be issued by the affected department **and a copy of the** form will be kept on file by the issuing Department.

**Exception:** Warehouse delivery drivers/shipping drivers are allowed to take pictures of their loads without a camera pass provided that the refinery units are not in the background & use is approved by a Marathon Representative.

2. **Must carry the Approval Form while using the camera inside the Refinery.**
3. Before being distributed outside the Company, all photos/videos taken by the contractor must be reviewed by the Department Manager or his/her designee. Photos to be used outside the Company for publications, public presentations, etc. must be provided to the local Human Resources Department who will obtain Corporate Public Affairs approval for their use.

**NOTE:** Anyone in violation of this procedure will have their photographic equipment confiscated and will be escorted out of the refinery until a determination is made as to the appropriateness of the photos.



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## Appendix C - Electric & Instrument Shop Locked Vehicle Program

### Purpose:

In an effort to safeguard tools, protect company property, and ensure shop vehicles are available when responding to off-shift equipment failure call outs, permission to park and lock Electric and Instrument shop vehicles in designated locations has been granted by IRD Management Staff. All other vehicles within the refinery fence must be unlocked with keys in the ignition.

### Details:

This document details the Electric & Instrument Shop locked vehicle program. Vehicles belonging to the Electric and Instrument shop may be locked at the end of each shift given all the following provisions:

- Vehicles are parked in any of the designated locked vehicle parking spaces per the drawing below.
- Vehicle keys are located within the designed key storage space within the Craft Shops.
- Vehicle number of vehicle being locked is listed on the Locked Vehicle List (LVL).
- Locked Vehicle List (LVL) has been submitted to the Operations Shift Foreman.

Failure to abide by the provisions in this written program may result in the revocation of the same.

**Special Note:** HVAC Shop vehicles are included in this program due to the fact that they contain canisters of refrigerant which must be controlled and can only be handled by licensed personnel.

### Enforcement:

Responsibility for enforcement of this policy resides with the Electric and Instrument Shop Foremen. Checks should be made on a periodic basis to ensure this policy is being followed by shop personnel.

### Notice:

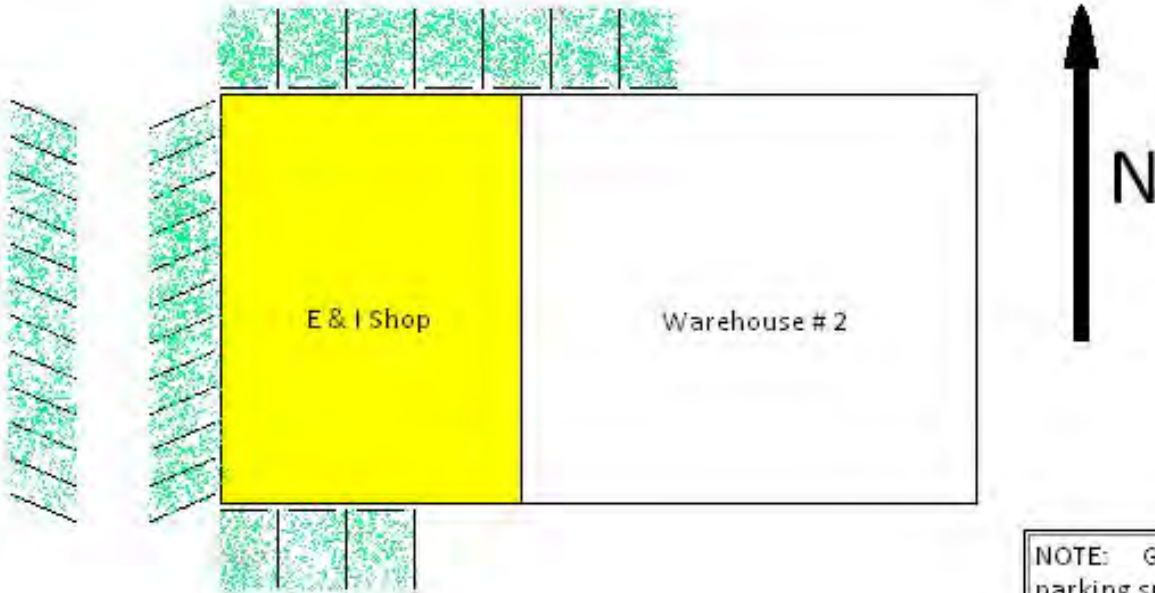
Specific general maintenance vehicles have been designated for use by operations personnel to shuttle workers to and from their complexes. It is the responsibility of the Maintenance Department to see that these vehicles are located in their designated parking spaces, at the designated time, for use by operations personnel. If the vehicles cannot be in their designated spaces at the designated time for whatever reason, the Operations Shift Foreman is to be notified so provisions can be made.

### Attachment:

See the diagram on the next page for designated locked vehicle parking spaces.

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NOTE: Green shaded parking spaces indicate locations of locked CRAFT vehicles during Maintenance Department off-shift hours. Vehicle keys are located at designated areas inside CRAFT shops. Shift Foreman can obtain keys in an emergency.



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## Appendix D - Designated Kitchen Areas

### **General**

96B-1019, Main Office West Coffee Shop & Kitchen  
 96B-1108, Main Office East Basement Kitchen & Coffee Bars 1<sup>st</sup> & 2<sup>nd</sup> floors  
 96B-1116, Security Operations Center Kitchen Area

### **Environmental & Safety**

96B-1014, #1 Fire House Kitchen Area  
 96B-1079, West Receiving Gate Security Office, Coffee Bar  
 96B-2002, "C" Gate Guard Shack Coffee Bar  
 96B-2003, "SW" Gate Guard Shack Coffee Bar  
 96B-5034, Fire Field Trailer Kitchen Area  
 96B-5176, Environmental Trailer Break Area  
 96B-2179, Safety Office Trailer Kitchen Area

### **PDU**

96B-1105, Laboratory Kitchen Area  
 96B-5077, Propane Loading Operator Trailer Break Area  
 96B-1123, PDU Operator Control Room Kitchen Area

### **Operations**

96B-1004, Central Control Room Kitchen Area & CCR Annex Break Area  
 96B-1120, CX-1 Operations Shelter BRM Kitchen Area  
 96B-1071, CX-2 Operations Shelter 2<sup>nd</sup> floor (LTBA) Kitchen area  
 96B-1111, Cx- 3 Operations Shelter BRM Kitchen Area  
 96B-1125, CX-5 Operations Shelter BRM Kitchen Area  
 96B-1122, CX-6 Operator Shelter BRM Kitchen Area  
 96B-2106, Coker Operator Trailer Break Area  
 96B-1127 MPL Robinson Wabash Station Kitchen Area  
 96B-1113, CX-7 Operations Control Room (GDU) Kitchen Area  
 96B-1011, Filter Press Building Kitchen Area  
 96B-1082, Waste Water Treatment Trailer Kitchen Area

### **Maintenance, Warehouse, and Contractors**

96B-1023, Machine Shop Break Room & Garage Kitchen Area  
 96B-1025, E&I Shop Kitchen Area  
 96B-1015, Main Warehouse Kitchen Area  
 96B-1071, Warehouse #3, 1<sup>st</sup> floor (LTBA) Kitchen Area  
 96B-1050, Carpenter Shop Kitchen Area  
 96B-1027 Communication Building  
 96B-5099, TAR Offices/Training Trailer Coffee Bar  
 96B-1003, Inspection CM Group Annex Coffee Bar  
 96B-1068, New Maintenance Building Kitchen Area (one in each of the five Area Rooms)  
 96B-5209, TAR Planning Trailer Kitchen Area  
 96B-1107, Area Maintenance Shop Break Room  
 Contractor Break Trailers or Office Trailers  
 Temporary Buildings for TAR or Project Use

Marathon Petroleum Company LP			
<b>Safety Procedure #12 GENERAL SAFETY RULES</b>	Document No. <b>1012</b>	Page <b>Page 26 of 31</b>	Revision No. <b>37</b>
	Original Issue Date 4/98	Revision Date <b>07-26-2019</b>	Next Revision Date <b>10-1-2020</b>
Document Custodian: <b>Safety</b>	Content Category: <b>Policy, Standard, Procedure or Guideline - General</b> Content Type: <b>Policy - Operating</b> Retention Period: <b>ACT + 10 years</b>		

**Appendix E  
Personal Protective Equipment Reference Guide (Minimum Requirements)- Physical Hazards**

Electrical	High Noise > 85 dBA	Falling Objects	Flying Objects	Foot Hazards	Chemical Splashes	Heights over 4 feet	Flash Fires	Thermal Burns	Handling Sharp Objects	Welding, brazing, cutting	Soldering
X											
		X	X								
		X	X		X						
	X										
		X									
				X							
				X							
						X					
							X				
								X			
								X			
			X		X				X		
X											
										X	
											X

**Personal Protective Equipment Reference Guide (Minimum Requirements)- Health Hazards**

Sulfuric Acid	HF Acid	Ammonia	Perchloroethylene / Ethylene Dichloride (chloriding agents)	Caustic (Sodium Hydroxide)	H <sub>2</sub> S	Sulfur Dioxide	Nitrogen (used for purging)	Welding Fumes	Carbon Monoxide	Asbestos	Hydrocarbons containing <10%Benzene	Hydrocarbons containing >10%Be
X			X	X								
X			X	X								
X		X	X	X								
	X											
					X							
					X							

Marathon Petroleum Company LP			
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	Original Issue Date 4/98	Revision Date <b>07-26-2019</b>	Next Revision Date <b>10-1-2020</b>
Document Custodian: <b>Safety</b>	Content Category: <b>Policy, Standard, Procedure or Guideline - General</b> Content Type: <b>Policy - Operating</b> Retention Period: <b>ACT + 10 years</b>		

							X					
		X										
X			X	X								
						X						
						X						
									X			
									X			
											X	
											X	X
											X	X
											X	X
												X

Marathon Petroleum Company LP			
<b>Safety Procedure #12 GENERAL SAFETY RULES</b>	Document No. <b>1012</b>	Page <b>Page 28 of 31</b>	Revision No. <b>37</b>
	Original Issue Date 4/98	Revision Date <b>07-26-2019</b>	Next Revision Date <b>10-1-2020</b>
Document Custodian: <b>Safety</b>	Content Category: <b>Policy, Standard, Procedure or Guideline - General</b> Content Type: <b>Policy - Operating</b> Retention Period: <b>ACT + 10 years</b>		

## Appendix F

### Impact Hazard Table

Work Group	Potential Impact Hazards
Operations	Disconnecting/Connecting railcars, using valve wrenches in tight quarters, hand wrenching flange bolts, hoisting materials, running impact guns, exposure to pinch points, etc.
Maintenance	Installing/Removing blinds, rebar work, lifting/setting pumps, hand wrenching flange bolts, hoisting materials, assembling/disassembling rigging, installing/removing piping, impact gun tasks, working with hammers, etc.
Contractor	Iron work, building scaffolding, rebar work, lifting/setting pumps, hand wrenching flange bolts, hoisting materials, assembling/disassembling rigging, installing/removing piping, impact gun tasks, working with hammers, etc.

**NOTE:** The list of hazards in the table above does not cover every situation in which personnel may be exposed to impact hazards. For tasks with the potential of impact hazards, gloves with impact protection to the back of the hand and full length of the fingers are to be worn.

Marathon Petroleum Company LP			
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Document Custodian: <b>Safety</b>	Content Category: <b>Policy, Standard, Procedure or Guideline - General</b> Content Type: <b>Policy - Operating</b> Retention Period: <b>ACT + 10 years</b>		

## Appendix G

### [IRD PPE Matrix](#)



Marathon Petroleum Company LP			
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	Original Issue Date 4/98	Revision Date <b>07-26-2019</b>	Next Revision Date <b>10-1-2020</b>
Document Custodian: <b>Safety</b>	Content Category: <b>Policy, Standard, Procedure or Guideline - General</b> Content Type: <b>Policy - Operating</b> Retention Period: <b>ACT + 10 years</b>		

## Appendix H – Electronic Device Approval Process

Marathon or Contractor personnel who have a legitimate business reason to use a personal device in a “Restricted” must have that device approved by a Department Manager. The Electronic Device Approval Form must be carried on your person whenever you are carrying or using the personal device in a restricted area.

Illinois Refining Division Electronic Device Approval
Issued To: _____ Company: _____ Device: <input type="checkbox"/> Cell Phone <input type="checkbox"/> Tablet <input type="checkbox"/> Other: _____ Business Purpose: _____
Approval (Must be a department manager) Name: _____ Signature: _____

### Electronic Device Approval Sticker

After completing the Approval Form above and in order to obtain an MPC Refining Approval Sticker for their device, the Contract Company must provide documentation that their device and/or device w/ case meets all the minimum requirements listed in Section II. B.2. to the Safety Supervisor.



Marathon Petroleum Company LP			
<b>Safety Procedure #12 GENERAL SAFETY RULES</b>	Document No. <b>1012</b>	Page <b>Page 31 of 31</b>	Revision No. <b>37</b>
	Original Issue Date <b>4/98</b>	Revision Date <b>07-26-2019</b>	Next Revision Date <b>10-1-2020</b>
Document Custodian: <b>Safety</b>	Content Category: <b>Policy, Standard, Procedure or Guideline - General</b> Content Type: <b>Policy - Operating</b> Retention Period: <b>ACT + 10 years</b>		

## Appendix I – PEP2 Device Evaluation Form

ANSI Section #	ANSI/ISA-12.12.03-2011 "PEP2" Requirements Applicable to Case	Does the Phone/Case Combination meet the intent of ANSI/ISA.12.12.03-2011 "PEP2"?	Do Phone (Model #) Specifications Meet Requirement?
6.1(b)	Radio Frequency Energy Transmission limited in accordance w/ 8.3.		
6.1(c)	No provisions for forced ventilation.		
6.1(d)	No sparks visible in normal operation.		
6.1(e)	No excessive temperatures in normal operation (>60 ℱ or 140 ℃).		
6.1(f)	No camera flash unless it can be disabled.		
6.1(g)	No motors unless it can be demonstrated the motor incorporates non-arcing technology.		
6.3(c)	Body-worn or Hand-held.		
6.3(a)	Powered by one or more cells, batteries, or photovoltaic cells.		
6.3(f)	Power switch in accordance with 8.2 (no power on/off switches w/ contact that directly interrupt battery current).		
6.1(h)	No visible damage.		
6.3(b)	Cell or battery secured so it will not fall out in the drop test as described of 8.1 (2 meter drop).		
6.3(e)	Exposed terminals (i.e., battery charging terminals) are either recessed or diode protected to prevent a discharge caused by an accidental shorting of these terminals.		
6.3(g)	No damage that exposes the electrical/electronic circuitry as a result of the drop test described in 8.1 (2 meter drop).		
ANSI Section #	ANSI/ISA-12.12.03-2011 Other "PEP2" Requirements	Does the Phone/Case Combination meet the intent of ANSI/ISA.12.12.03-2011 "PEP2"?	How will we meet these specifications?
6.1(a)	There must be no available listed apparatus suitable for the area classification & capable of performing the intended function (See section 4.12 definition of "listed"). OSHA defines "listed" in 1910.399 as "of a kind mentioned in a list that is published by a nationally recognized laboratory that makes periodic inspection of the production of such equipment, and states that such equipment meets the nationally recognized standards or has been tested & found		
6.3(d)	No external electrical connections or wired accessories are used in the hazardous classified location.		
7.1	A process of administrative control & training is necessary to ensure that portable products do not present an unacceptable risk of ignition when used in hazardous classified areas.		
7.2	The owner/operator of the hazardous classified location should establish a process of inspection in which a qualified person establishes that particular products can be accepted as PEP2.		
7.2	Supporting documentation for PEP2 evaluations should be maintained for the life of the use of those products. The documentation should include information for the the products such as reference number or code, product manufacturer and model, owner name, approver name, and date approved or equivalent information.		
g	Accepted PEP2 products should be marked "PEP2, evaluating company name, and supporting documentation reference		

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**Appendix B**

**Water Chemistry Laboratory Report  
19 September 2022**

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October 11, 2022

Ms. Julie Holscher  
Marathon Petroleum Company (Robinson IL)  
100 Marathon Ave.  
Robinson, IL 62454

RE: Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

Dear Ms. Holscher:

Enclosed are the analytical results for sample(s) received by the laboratory on September 20, 2022. The results relate only to the samples included in this report. Results reported herein conform to the applicable TNI/NELAC Standards and the laboratory's Quality Manual, where applicable, unless otherwise noted in the body of the report.

The test results provided in this final report were generated by each of the following laboratories within the Pace Network:

- Pace Analytical Services - Indianapolis

If you have any questions concerning this report, please feel free to contact me.

Sincerely,



Kenneth Hunt  
kenneth.hunt@pacelabs.com  
(317)228-3100  
Project Manager

Enclosures

cc: Mr. Patrick Beabout, Marathon Robinson Refinery  
Ms. Sara Clough, Marathon Robinson Refinery  
Mr. Michael Elliott, Marathon Robinson  
Ms. Emily Gullett, Marathon Robinson Refinery  
Mr. Douglas McNary, Marathon Petroleum (Robinson IL)  
Mr. Dillon O'Kelly, Marathon Robinson  
Jared Ridge, Marathon Robinson



## REPORT OF LABORATORY ANALYSIS

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### CERTIFICATIONS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

---

**Pace Analytical Services Indianapolis**

7726 Moller Road, Indianapolis, IN 46268

Illinois Accreditation #: 200074

Indiana Drinking Water Laboratory #: C-49-06

Kansas/TNI Certification #: E-10177

Kentucky UST Agency Interest #: 80226

Kentucky WW Laboratory ID #: 98019

Michigan Drinking Water Laboratory #9050

Ohio VAP Certified Laboratory #: CL0065

Oklahoma Laboratory #: 9204

Texas Certification #: T104704355

Wisconsin Laboratory #: 999788130

USDA Soil Permit #: P330-19-00257

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### REPORT OF LABORATORY ANALYSIS

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### SAMPLE SUMMARY

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Lab ID	Sample ID	Matrix	Date Collected	Date Received
50326308001	20-CON-19SEP2022	Water	09/19/22 12:45	09/20/22 12:15
50326308002	20-UPS-19SEP2022	Water	09/19/22 16:35	09/20/22 12:15
50326308003	20-EFF-19SEP2022	Water	09/19/22 15:35	09/20/22 12:15
50326308004	20-DNS-19SEP2022	Water	09/19/22 12:10	09/20/22 12:15
50326308005	30-CON-19SEP2022	Water	09/19/22 16:05	09/20/22 12:15
50326308006	30-UPS-19SEP2022	Water	09/19/22 13:30	09/20/22 12:15
50326308007	30-EFF-19SEP2022	Water	09/19/22 16:20	09/20/22 12:15
50326308008	30-DNS-19SEP2022	Water	09/19/22 15:50	09/20/22 12:15

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**SAMPLE ANALYTE COUNT**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
50326308001	20-CON-19SEP2022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	TKG	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50326308002	20-UPS-19SEP2022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	TKG	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50326308003	20-EFF-19SEP2022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	TKG	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50326308004	20-DNS-19SEP2022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	TKG	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50326308005	30-CON-19SEP2022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	TKG	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50326308006	30-UPS-19SEP2022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I

**REPORT OF LABORATORY ANALYSIS**

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**SAMPLE ANALYTE COUNT**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
50326308007	30-EFF-19SEP2022	EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	TKG	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
		EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	TKG	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50326308008	30-DNS-19SEP2022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	TKG	32	PASI-I
		EPA 335.4	ZM	1	PASI-I

PASI-I = Pace Analytical Services - Indianapolis

**REPORT OF LABORATORY ANALYSIS**

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-CON-19SEP2022 Lab ID: 50326308001 Collected: 09/19/22 12:45 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>608.3 PCB</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
PCB-1016 (Aroclor 1016)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:00	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:00	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:00	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:00	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:00	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:00	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.098	0.050	1	09/22/22 23:01	09/26/22 21:00	11096-82-5	
<b>Surrogates</b>									
Tetrachloro-m-xylene (S)	55	%	1-123		1	09/22/22 23:01	09/26/22 21:00	877-09-8	
<b>608.3 Pesticides</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
Aldrin	ND	ug/L	0.049	0.0088	1	09/22/22 23:01	10/10/22 15:22	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.049	0.011	1	09/22/22 23:01	10/10/22 15:22	319-84-6	
beta-BHC	ND	ug/L	0.049	0.014	1	09/22/22 23:01	10/10/22 15:22	319-85-7	
delta-BHC	ND	ug/L	0.049	0.013	1	09/22/22 23:01	10/10/22 15:22	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.049	0.012	1	09/22/22 23:01	10/10/22 15:22	58-89-9	
Chlordane (Technical)	ND	ug/L	0.49	0.26	1	09/22/22 23:01	10/10/22 15:22	57-74-9	
4,4'-DDD	ND	ug/L	0.098	0.024	1	09/22/22 23:01	10/10/22 15:22	72-54-8	
4,4'-DDE	ND	ug/L	0.098	0.018	1	09/22/22 23:01	10/10/22 15:22	72-55-9	
4,4'-DDT	ND	ug/L	0.098	0.035	1	09/22/22 23:01	10/10/22 15:22	50-29-3	
Dieldrin	ND	ug/L	0.098	0.022	1	09/22/22 23:01	10/10/22 15:22	60-57-1	
Endosulfan I	ND	ug/L	0.049	0.012	1	09/22/22 23:01	10/10/22 15:22	959-98-8	
Endosulfan II	ND	ug/L	0.098	0.025	1	09/22/22 23:01	10/10/22 15:22	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.098	0.021	1	09/22/22 23:01	10/10/22 15:22	1031-07-8	
Endrin	ND	ug/L	0.098	0.026	1	09/22/22 23:01	10/10/22 15:22	72-20-8	
Endrin aldehyde	ND	ug/L	0.098	0.025	1	09/22/22 23:01	10/10/22 15:22	7421-93-4	
Heptachlor	ND	ug/L	0.049	0.0098	1	09/22/22 23:01	10/10/22 15:22	76-44-8	H7,L2
Heptachlor epoxide	ND	ug/L	0.049	0.011	1	09/22/22 23:01	10/10/22 15:22	1024-57-3	
Toxaphene	ND	ug/L	0.98	0.35	1	09/22/22 23:01	10/10/22 15:22	8001-35-2	
<b>Surrogates</b>									
Decachlorobiphenyl (S)	45	%	1-140		1	09/22/22 23:01	10/10/22 15:22	2051-24-3	
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Antimony	ND	mg/L	0.0010	0.00013	1	09/28/22 08:00	10/03/22 14:04	7440-36-0	
Arsenic	<b>0.00038J</b>	mg/L	0.0010	0.00011	1	09/28/22 08:00	10/03/22 14:04	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	09/28/22 08:00	10/03/22 14:04	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	09/28/22 08:00	10/03/22 14:04	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	09/28/22 08:00	10/03/22 14:04	7440-47-3	
Copper	<b>0.0043</b>	mg/L	0.0010	0.00037	1	09/28/22 08:00	10/03/22 14:04	7440-50-8	
Lead	ND	mg/L	0.0010	0.000080	1	09/28/22 08:00	10/03/22 14:04	7439-92-1	
Nickel	<b>0.0012</b>	mg/L	0.00050	0.00039	1	09/28/22 08:00	10/03/22 14:04	7440-02-0	
Selenium	<b>0.0020</b>	mg/L	0.0010	0.00035	1	09/28/22 08:00	10/03/22 14:04	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	09/28/22 08:00	10/03/22 14:04	7440-22-4	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-CON-19SEP2022 Lab ID: 50326308001 Collected: 09/19/22 12:45 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	ND	mg/L	0.0010	0.000073	1	09/28/22 08:00	10/03/22 14:04	7440-28-0	
Zinc	0.051	mg/L	0.0030	0.0010	1	09/28/22 08:00	10/03/22 14:04	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	09/26/22 20:21	09/27/22 11:53	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.6	1.7	1	09/22/22 19:22	09/23/22 16:54	83-32-9	
Acenaphthylene	ND	ug/L	9.6	1.8	1	09/22/22 19:22	09/23/22 16:54	208-96-8	
Anthracene	ND	ug/L	9.6	1.8	1	09/22/22 19:22	09/23/22 16:54	120-12-7	
Benzdine	ND	ug/L	48.1	5.8	1	09/22/22 19:22	09/23/22 16:54	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.6	1.6	1	09/22/22 19:22	09/23/22 16:54	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.6	2.2	1	09/22/22 19:22	09/23/22 16:54	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.6	2.5	1	09/22/22 19:22	09/23/22 16:54	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.6	2.1	1	09/22/22 19:22	09/23/22 16:54	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.6	2.5	1	09/22/22 19:22	09/23/22 16:54	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.6	2.8	1	09/22/22 19:22	09/23/22 16:54	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.6	3.6	1	09/22/22 19:22	09/23/22 16:54	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.2	2.9	1	09/22/22 19:22	09/23/22 16:54	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.6	3.0	1	09/22/22 19:22	09/23/22 16:54	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.6	2.1	1	09/22/22 19:22	09/23/22 16:54	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.6	3.5	1	09/22/22 19:22	09/23/22 16:54	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.6	2.5	1	09/22/22 19:22	09/23/22 16:54	91-58-7	
2-Chlorophenol	ND	ug/L	9.6	2.2	1	09/22/22 19:22	09/23/22 16:54	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.6	2.6	1	09/22/22 19:22	09/23/22 16:54	7005-72-3	
Chrysene	ND	ug/L	9.6	1.8	1	09/22/22 19:22	09/23/22 16:54	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.6	2.5	1	09/22/22 19:22	09/23/22 16:54	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.6	2.6	1	09/22/22 19:22	09/23/22 16:54	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.6	2.7	1	09/22/22 19:22	09/23/22 16:54	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.6	2.8	1	09/22/22 19:22	09/23/22 16:54	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.2	3.0	1	09/22/22 19:22	09/23/22 16:54	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.6	2.2	1	09/22/22 19:22	09/23/22 16:54	120-83-2	
Diethylphthalate	ND	ug/L	9.6	3.0	1	09/22/22 19:22	09/23/22 16:54	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.6	2.2	1	09/22/22 19:22	09/23/22 16:54	105-67-9	
Dimethylphthalate	ND	ug/L	9.6	2.2	1	09/22/22 19:22	09/23/22 16:54	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.6	3.2	1	09/22/22 19:22	09/23/22 16:54	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	48.1	7.8	1	09/22/22 19:22	09/23/22 16:54	534-52-1	
2,4-Dinitrophenol	ND	ug/L	48.1	5.0	1	09/22/22 19:22	09/23/22 16:54	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.6	2.3	1	09/22/22 19:22	09/23/22 16:54	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.6	2.7	1	09/22/22 19:22	09/23/22 16:54	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.6	7.1	1	09/22/22 19:22	09/23/22 16:54	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.6	2.0	1	09/22/22 19:22	09/23/22 16:54	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.8	4.0	1	09/22/22 19:22	09/23/22 16:54	117-81-7	

**REPORT OF LABORATORY ANALYSIS**

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-CON-19SEP2022 Lab ID: 50326308001 Collected: 09/19/22 12:45 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	9.6	1.8	1	09/22/22 19:22	09/23/22 16:54	206-44-0	
Fluorene	ND	ug/L	9.6	1.9	1	09/22/22 19:22	09/23/22 16:54	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.6	3.7	1	09/22/22 19:22	09/23/22 16:54	87-68-3	
Hexachlorobenzene	ND	ug/L	9.6	3.2	1	09/22/22 19:22	09/23/22 16:54	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.2	4.4	1	09/22/22 19:22	09/23/22 16:54	77-47-4	N2
Hexachloroethane	ND	ug/L	9.6	3.3	1	09/22/22 19:22	09/23/22 16:54	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.6	2.5	1	09/22/22 19:22	09/23/22 16:54	193-39-5	
Isophorone	ND	ug/L	9.6	1.9	1	09/22/22 19:22	09/23/22 16:54	78-59-1	
Naphthalene	ND	ug/L	9.6	2.3	1	09/22/22 19:22	09/23/22 16:54	91-20-3	
Nitrobenzene	ND	ug/L	9.6	3.0	1	09/22/22 19:22	09/23/22 16:54	98-95-3	
2-Nitrophenol	ND	ug/L	9.6	4.0	1	09/22/22 19:22	09/23/22 16:54	88-75-5	
4-Nitrophenol	ND	ug/L	48.1	4.9	1	09/22/22 19:22	09/23/22 16:54	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.2	3.4	1	09/22/22 19:22	09/23/22 16:54	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.6	2.8	1	09/22/22 19:22	09/23/22 16:54	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.6	2.1	1	09/22/22 19:22	09/23/22 16:54	86-30-6	
Pentachlorophenol	ND	ug/L	48.1	6.5	1	09/22/22 19:22	09/23/22 16:54	87-86-5	
Phenanthrene	ND	ug/L	9.6	1.8	1	09/22/22 19:22	09/23/22 16:54	85-01-8	
Phenol	ND	ug/L	9.6	1.2	1	09/22/22 19:22	09/23/22 16:54	108-95-2	
Pyrene	ND	ug/L	9.6	1.9	1	09/22/22 19:22	09/23/22 16:54	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.6	3.5	1	09/22/22 19:22	09/23/22 16:54	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.6	2.6	1	09/22/22 19:22	09/23/22 16:54	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	52	%	9-74		1	09/22/22 19:22	09/23/22 16:54	367-12-4	
Phenol-d5 (S)	37	%	8-424		1	09/22/22 19:22	09/23/22 16:54	4165-62-2	
Nitrobenzene-d5 (S)	81	%	15-314		1	09/22/22 19:22	09/23/22 16:54	4165-60-0	
2-Fluorobiphenyl (S)	78	%	32-92		1	09/22/22 19:22	09/23/22 16:54	321-60-8	
2,4,6-Tribromophenol (S)	85	%	27-125		1	09/22/22 19:22	09/23/22 16:54	118-79-6	
p-Terphenyl-d14 (S)	87	%	8-146		1	09/22/22 19:22	09/23/22 16:54	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	3.3	1		09/21/22 13:46	107-02-8	L1
Acrylonitrile	ND	ug/L	100	0.50	1		09/21/22 13:46	107-13-1	
Benzene	ND	ug/L	5.0	0.11	1		09/21/22 13:46	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.11	1		09/21/22 13:46	75-27-4	
Bromoform	ND	ug/L	5.0	0.036	1		09/21/22 13:46	75-25-2	
Bromomethane	ND	ug/L	5.0	0.19	1		09/21/22 13:46	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.065	1		09/21/22 13:46	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.098	1		09/21/22 13:46	108-90-7	
Chloroethane	ND	ug/L	5.0	0.17	1		09/21/22 13:46	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	0.37	1		09/21/22 13:46	110-75-8	
Chloroform	ND	ug/L	4.8	0.15	1		09/21/22 13:46	67-66-3	
Chloromethane	ND	ug/L	5.0	0.16	1		09/21/22 13:46	74-87-3	
Dibromochloromethane	0.56J	ug/L	5.0	0.041	1		09/21/22 13:46	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.10	1		09/21/22 13:46	75-34-3	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

**Sample: 20-CON-19SEP2022**      **Lab ID: 50326308001**      Collected: 09/19/22 12:45      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.12	1		09/21/22 13:46	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.13	1		09/21/22 13:46	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.13	1		09/21/22 13:46	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.12	1		09/21/22 13:46	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 13:46	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 13:46	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.082	1		09/21/22 13:46	100-41-4	
Methylene Chloride	ND	ug/L	5.0	1.6	1		09/21/22 13:46	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.090	1		09/21/22 13:46	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.16	1		09/21/22 13:46	127-18-4	
Toluene	ND	ug/L	5.0	0.11	1		09/21/22 13:46	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.097	1		09/21/22 13:46	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.11	1		09/21/22 13:46	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.12	1		09/21/22 13:46	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.11	1		09/21/22 13:46	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	95	%	91-114		1		09/21/22 13:46	1868-53-7	
4-Bromofluorobenzene (S)	98	%	85-120		1		09/21/22 13:46	460-00-4	
Toluene-d8 (S)	100	%	85-117		1		09/21/22 13:46	2037-26-5	

**335.4 Cyanide, Total**

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	ND	mg/L	0.0050	0.0018	1	09/26/22 11:46	09/27/22 17:37	57-12-5	
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**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-UPS-19SEP2022 Lab ID: 50326308002 Collected: 09/19/22 16:35 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>608.3 PCB</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
PCB-1016 (Aroclor 1016)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:14	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:14	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:14	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:14	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:14	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 21:14	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.098	0.050	1	09/22/22 23:01	09/26/22 21:14	11096-82-5	
<b>Surrogates</b>									
Tetrachloro-m-xylene (S)	42	%	1-123		1	09/22/22 23:01	09/26/22 21:14	877-09-8	
<b>608.3 Pesticides</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
Aldrin	ND	ug/L	0.049	0.0088	1	09/22/22 23:01	10/10/22 15:35	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.049	0.011	1	09/22/22 23:01	10/10/22 15:35	319-84-6	
beta-BHC	ND	ug/L	0.049	0.014	1	09/22/22 23:01	10/10/22 15:35	319-85-7	
delta-BHC	ND	ug/L	0.049	0.013	1	09/22/22 23:01	10/10/22 15:35	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.049	0.012	1	09/22/22 23:01	10/10/22 15:35	58-89-9	
Chlordane (Technical)	ND	ug/L	0.49	0.26	1	09/22/22 23:01	10/10/22 15:35	57-74-9	
4,4'-DDD	ND	ug/L	0.098	0.024	1	09/22/22 23:01	10/10/22 15:35	72-54-8	
4,4'-DDE	ND	ug/L	0.098	0.018	1	09/22/22 23:01	10/10/22 15:35	72-55-9	
4,4'-DDT	ND	ug/L	0.098	0.035	1	09/22/22 23:01	10/10/22 15:35	50-29-3	
Dieldrin	ND	ug/L	0.098	0.022	1	09/22/22 23:01	10/10/22 15:35	60-57-1	
Endosulfan I	ND	ug/L	0.049	0.012	1	09/22/22 23:01	10/10/22 15:35	959-98-8	
Endosulfan II	ND	ug/L	0.098	0.025	1	09/22/22 23:01	10/10/22 15:35	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.098	0.021	1	09/22/22 23:01	10/10/22 15:35	1031-07-8	
Endrin	ND	ug/L	0.098	0.026	1	09/22/22 23:01	10/10/22 15:35	72-20-8	
Endrin aldehyde	ND	ug/L	0.098	0.025	1	09/22/22 23:01	10/10/22 15:35	7421-93-4	
Heptachlor	ND	ug/L	0.049	0.0098	1	09/22/22 23:01	10/10/22 15:35	76-44-8	H7,L2
Heptachlor epoxide	ND	ug/L	0.049	0.011	1	09/22/22 23:01	10/10/22 15:35	1024-57-3	
Toxaphene	ND	ug/L	0.98	0.35	1	09/22/22 23:01	10/10/22 15:35	8001-35-2	
<b>Surrogates</b>									
Decachlorobiphenyl (S)	62	%	1-140		1	09/22/22 23:01	10/10/22 15:35	2051-24-3	
<b>200.8 Metals, Total ICMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Antimony	0.00032J	mg/L	0.0010	0.00013	1	09/28/22 08:00	10/03/22 14:08	7440-36-0	
Arsenic	0.0014	mg/L	0.0010	0.00011	1	09/28/22 08:00	10/03/22 14:08	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	09/28/22 08:00	10/03/22 14:08	7440-41-7	
Cadmium	0.00082	mg/L	0.00020	0.000034	1	09/28/22 08:00	10/03/22 14:08	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	09/28/22 08:00	10/03/22 14:08	7440-47-3	
Copper	0.0020	mg/L	0.0010	0.00037	1	09/28/22 08:00	10/03/22 14:08	7440-50-8	
Lead	0.00014J	mg/L	0.0010	0.000080	1	09/28/22 08:00	10/03/22 14:08	7439-92-1	
Nickel	0.0033	mg/L	0.00050	0.00039	1	09/28/22 08:00	10/03/22 14:08	7440-02-0	
Selenium	0.0010	mg/L	0.0010	0.00035	1	09/28/22 08:00	10/03/22 14:08	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	09/28/22 08:00	10/03/22 14:08	7440-22-4	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-UPS-19SEP2022 Lab ID: 50326308002 Collected: 09/19/22 16:35 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	ND	mg/L	0.0010	0.000073	1	09/28/22 08:00	10/03/22 14:08	7440-28-0	
Zinc	0.025	mg/L	0.0030	0.0010	1	09/28/22 08:00	10/03/22 14:08	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/03/22 09:26	10/03/22 13:04	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	10.0	1.8	1	09/22/22 19:22	09/23/22 17:10	83-32-9	
Acenaphthylene	ND	ug/L	10.0	1.9	1	09/22/22 19:22	09/23/22 17:10	208-96-8	
Anthracene	ND	ug/L	10.0	1.9	1	09/22/22 19:22	09/23/22 17:10	120-12-7	
Benzdine	ND	ug/L	50.0	6.0	1	09/22/22 19:22	09/23/22 17:10	92-87-5	
Benzo(a)anthracene	ND	ug/L	10.0	1.7	1	09/22/22 19:22	09/23/22 17:10	56-55-3	
Benzo(a)pyrene	ND	ug/L	10.0	2.3	1	09/22/22 19:22	09/23/22 17:10	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	10.0	2.6	1	09/22/22 19:22	09/23/22 17:10	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	10.0	2.2	1	09/22/22 19:22	09/23/22 17:10	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	10.0	2.6	1	09/22/22 19:22	09/23/22 17:10	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	10.0	2.9	1	09/22/22 19:22	09/23/22 17:10	101-55-3	
Butylbenzylphthalate	ND	ug/L	10.0	3.7	1	09/22/22 19:22	09/23/22 17:10	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	20.0	3.0	1	09/22/22 19:22	09/23/22 17:10	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	10.0	3.1	1	09/22/22 19:22	09/23/22 17:10	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	10.0	2.2	1	09/22/22 19:22	09/23/22 17:10	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	10.0	3.6	1	09/22/22 19:22	09/23/22 17:10	108-60-1	
2-Chloronaphthalene	ND	ug/L	10.0	2.6	1	09/22/22 19:22	09/23/22 17:10	91-58-7	
2-Chlorophenol	ND	ug/L	10.0	2.3	1	09/22/22 19:22	09/23/22 17:10	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	10.0	2.7	1	09/22/22 19:22	09/23/22 17:10	7005-72-3	
Chrysene	ND	ug/L	10.0	1.9	1	09/22/22 19:22	09/23/22 17:10	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	10.0	2.6	1	09/22/22 19:22	09/23/22 17:10	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	10.0	2.7	1	09/22/22 19:22	09/23/22 17:10	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	10.0	2.9	1	09/22/22 19:22	09/23/22 17:10	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	10.0	2.9	1	09/22/22 19:22	09/23/22 17:10	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	20.0	3.1	1	09/22/22 19:22	09/23/22 17:10	91-94-1	
2,4-Dichlorophenol	ND	ug/L	10.0	2.3	1	09/22/22 19:22	09/23/22 17:10	120-83-2	
Diethylphthalate	ND	ug/L	10.0	3.2	1	09/22/22 19:22	09/23/22 17:10	84-66-2	
2,4-Dimethylphenol	ND	ug/L	10.0	2.3	1	09/22/22 19:22	09/23/22 17:10	105-67-9	
Dimethylphthalate	ND	ug/L	10.0	2.3	1	09/22/22 19:22	09/23/22 17:10	131-11-3	
Di-n-butylphthalate	ND	ug/L	10.0	3.3	1	09/22/22 19:22	09/23/22 17:10	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	50.0	8.2	1	09/22/22 19:22	09/23/22 17:10	534-52-1	
2,4-Dinitrophenol	ND	ug/L	50.0	5.2	1	09/22/22 19:22	09/23/22 17:10	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	10.0	2.4	1	09/22/22 19:22	09/23/22 17:10	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	10.0	2.9	1	09/22/22 19:22	09/23/22 17:10	606-20-2	
Di-n-octylphthalate	ND	ug/L	10.0	7.3	1	09/22/22 19:22	09/23/22 17:10	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	10.0	2.1	1	09/22/22 19:22	09/23/22 17:10	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	5.0	4.2	1	09/22/22 19:22	09/23/22 17:10	117-81-7	

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

**Sample: 20-UPS-19SEP2022**      **Lab ID: 50326308002**      Collected: 09/19/22 16:35      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**625.1 MSSV**

Analytical Method: EPA 625.1      Preparation Method: EPA 625.1  
Pace Analytical Services - Indianapolis

Fluoranthene	ND	ug/L	10.0	1.8	1	09/22/22 19:22	09/23/22 17:10	206-44-0	
Fluorene	ND	ug/L	10.0	2.0	1	09/22/22 19:22	09/23/22 17:10	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	10.0	3.8	1	09/22/22 19:22	09/23/22 17:10	87-68-3	
Hexachlorobenzene	ND	ug/L	10.0	3.3	1	09/22/22 19:22	09/23/22 17:10	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	20.0	4.5	1	09/22/22 19:22	09/23/22 17:10	77-47-4	N2
Hexachloroethane	ND	ug/L	10.0	3.4	1	09/22/22 19:22	09/23/22 17:10	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	10.0	2.6	1	09/22/22 19:22	09/23/22 17:10	193-39-5	
Isophorone	ND	ug/L	10.0	1.9	1	09/22/22 19:22	09/23/22 17:10	78-59-1	
Naphthalene	ND	ug/L	10.0	2.4	1	09/22/22 19:22	09/23/22 17:10	91-20-3	
Nitrobenzene	ND	ug/L	10.0	3.1	1	09/22/22 19:22	09/23/22 17:10	98-95-3	
2-Nitrophenol	ND	ug/L	10.0	4.2	1	09/22/22 19:22	09/23/22 17:10	88-75-5	
4-Nitrophenol	ND	ug/L	50.0	5.1	1	09/22/22 19:22	09/23/22 17:10	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	20.0	3.5	1	09/22/22 19:22	09/23/22 17:10	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	10.0	3.0	1	09/22/22 19:22	09/23/22 17:10	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	10.0	2.2	1	09/22/22 19:22	09/23/22 17:10	86-30-6	
Pentachlorophenol	ND	ug/L	50.0	6.7	1	09/22/22 19:22	09/23/22 17:10	87-86-5	
Phenanthrene	ND	ug/L	10.0	1.9	1	09/22/22 19:22	09/23/22 17:10	85-01-8	
Phenol	ND	ug/L	10.0	1.2	1	09/22/22 19:22	09/23/22 17:10	108-95-2	
Pyrene	ND	ug/L	10.0	2.0	1	09/22/22 19:22	09/23/22 17:10	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	10.0	3.7	1	09/22/22 19:22	09/23/22 17:10	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	10.0	2.8	1	09/22/22 19:22	09/23/22 17:10	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	49	%	9-74		1	09/22/22 19:22	09/23/22 17:10	367-12-4	
Phenol-d5 (S)	36	%	8-424		1	09/22/22 19:22	09/23/22 17:10	4165-62-2	
Nitrobenzene-d5 (S)	83	%	15-314		1	09/22/22 19:22	09/23/22 17:10	4165-60-0	
2-Fluorobiphenyl (S)	74	%	32-92		1	09/22/22 19:22	09/23/22 17:10	321-60-8	
2,4,6-Tribromophenol (S)	89	%	27-125		1	09/22/22 19:22	09/23/22 17:10	118-79-6	
p-Terphenyl-d14 (S)	90	%	8-146		1	09/22/22 19:22	09/23/22 17:10	1718-51-0	

**624.1 Volatile Organics**

Analytical Method: EPA 624.1  
Pace Analytical Services - Indianapolis

Acrolein	ND	ug/L	50.0	3.3	1		09/21/22 14:15	107-02-8	L1
Acrylonitrile	ND	ug/L	100	0.50	1		09/21/22 14:15	107-13-1	
Benzene	ND	ug/L	5.0	0.11	1		09/21/22 14:15	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.11	1		09/21/22 14:15	75-27-4	
Bromoform	ND	ug/L	5.0	0.036	1		09/21/22 14:15	75-25-2	
Bromomethane	ND	ug/L	5.0	0.19	1		09/21/22 14:15	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.065	1		09/21/22 14:15	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.098	1		09/21/22 14:15	108-90-7	
Chloroethane	ND	ug/L	5.0	0.17	1		09/21/22 14:15	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	0.37	1		09/21/22 14:15	110-75-8	
Chloroform	ND	ug/L	4.8	0.15	1		09/21/22 14:15	67-66-3	
Chloromethane	ND	ug/L	5.0	0.16	1		09/21/22 14:15	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.041	1		09/21/22 14:15	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.10	1		09/21/22 14:15	75-34-3	

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

**Sample: 20-UPS-19SEP2022**      **Lab ID: 50326308002**      Collected: 09/19/22 16:35      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.12	1		09/21/22 14:15	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.13	1		09/21/22 14:15	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.13	1		09/21/22 14:15	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.12	1		09/21/22 14:15	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 14:15	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 14:15	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.082	1		09/21/22 14:15	100-41-4	
Methylene Chloride	ND	ug/L	5.0	1.6	1		09/21/22 14:15	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.090	1		09/21/22 14:15	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.16	1		09/21/22 14:15	127-18-4	
Toluene	ND	ug/L	5.0	0.11	1		09/21/22 14:15	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.097	1		09/21/22 14:15	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.11	1		09/21/22 14:15	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.12	1		09/21/22 14:15	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.11	1		09/21/22 14:15	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	96	%	91-114		1		09/21/22 14:15	1868-53-7	
4-Bromofluorobenzene (S)	100	%	85-120		1		09/21/22 14:15	460-00-4	
Toluene-d8 (S)	102	%	85-117		1		09/21/22 14:15	2037-26-5	
<b>335.4 Cyanide, Total</b>									
Analytical Method: EPA 335.4      Preparation Method: EPA 335.4									
Pace Analytical Services - Indianapolis									
Cyanide	ND	mg/L	0.0050	0.0018	1	09/26/22 11:46	09/27/22 17:37	57-12-5	

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-EFF-19SEP2022 Lab ID: 50326308003 Collected: 09/19/22 15:35 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>608.3 PCB</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
PCB-1016 (Aroclor 1016)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:28	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:28	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:28	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:28	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:28	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:28	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.10	0.051	1	09/22/22 23:01	09/26/22 21:28	11096-82-5	
<b>Surrogates</b>									
Tetrachloro-m-xylene (S)	55	%	1-123		1	09/22/22 23:01	09/26/22 21:28	877-09-8	
<b>608.3 Pesticides</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
Aldrin	ND	ug/L	0.050	0.0090	1	09/22/22 23:01	10/10/22 15:47	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.050	0.011	1	09/22/22 23:01	10/10/22 15:47	319-84-6	
beta-BHC	ND	ug/L	0.050	0.014	1	09/22/22 23:01	10/10/22 15:47	319-85-7	
delta-BHC	ND	ug/L	0.050	0.013	1	09/22/22 23:01	10/10/22 15:47	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.050	0.012	1	09/22/22 23:01	10/10/22 15:47	58-89-9	
Chlordane (Technical)	ND	ug/L	0.50	0.27	1	09/22/22 23:01	10/10/22 15:47	57-74-9	
4,4'-DDD	ND	ug/L	0.10	0.024	1	09/22/22 23:01	10/10/22 15:47	72-54-8	
4,4'-DDE	ND	ug/L	0.10	0.018	1	09/22/22 23:01	10/10/22 15:47	72-55-9	
4,4'-DDT	ND	ug/L	0.10	0.036	1	09/22/22 23:01	10/10/22 15:47	50-29-3	
Dieldrin	ND	ug/L	0.10	0.022	1	09/22/22 23:01	10/10/22 15:47	60-57-1	
Endosulfan I	ND	ug/L	0.050	0.012	1	09/22/22 23:01	10/10/22 15:47	959-98-8	
Endosulfan II	ND	ug/L	0.10	0.025	1	09/22/22 23:01	10/10/22 15:47	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.10	0.021	1	09/22/22 23:01	10/10/22 15:47	1031-07-8	
Endrin	ND	ug/L	0.10	0.027	1	09/22/22 23:01	10/10/22 15:47	72-20-8	
Endrin aldehyde	ND	ug/L	0.10	0.025	1	09/22/22 23:01	10/10/22 15:47	7421-93-4	
Heptachlor	ND	ug/L	0.050	0.010	1	09/22/22 23:01	10/10/22 15:47	76-44-8	H7,L2
Heptachlor epoxide	ND	ug/L	0.050	0.011	1	09/22/22 23:01	10/10/22 15:47	1024-57-3	
Toxaphene	ND	ug/L	1.0	0.36	1	09/22/22 23:01	10/10/22 15:47	8001-35-2	
<b>Surrogates</b>									
Decachlorobiphenyl (S)	38	%	1-140		1	09/22/22 23:01	10/10/22 15:47	2051-24-3	
<b>200.8 Metals, Total ICMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Antimony	0.0016	mg/L	0.0010	0.00013	1	09/28/22 08:00	10/03/22 14:12	7440-36-0	
Arsenic	0.0043	mg/L	0.0010	0.00011	1	09/28/22 08:00	10/03/22 14:12	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	09/28/22 08:00	10/03/22 14:12	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	09/28/22 08:00	10/03/22 14:12	7440-43-9	
Chromium	0.00067J	mg/L	0.0020	0.00063	1	09/28/22 08:00	10/03/22 14:12	7440-47-3	
Copper	0.0083	mg/L	0.0010	0.00037	1	09/28/22 08:00	10/03/22 14:12	7440-50-8	
Lead	ND	mg/L	0.0010	0.000080	1	09/28/22 08:00	10/03/22 14:12	7439-92-1	
Nickel	0.0052	mg/L	0.00050	0.00039	1	09/28/22 08:00	10/03/22 14:12	7440-02-0	
Selenium	0.11	mg/L	0.0010	0.00035	1	09/28/22 08:00	10/03/22 14:12	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	09/28/22 08:00	10/03/22 14:12	7440-22-4	

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-EFF-19SEP2022 Lab ID: 50326308003 Collected: 09/19/22 15:35 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	0.00024J	mg/L	0.0010	0.000073	1	09/28/22 08:00	10/03/22 14:12	7440-28-0	
Zinc	0.016	mg/L	0.0030	0.0010	1	09/28/22 08:00	10/03/22 14:12	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/03/22 09:26	10/03/22 13:06	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.8	1.8	1	09/22/22 19:22	09/23/22 17:26	83-32-9	
Acenaphthylene	ND	ug/L	9.8	1.8	1	09/22/22 19:22	09/23/22 17:26	208-96-8	
Anthracene	ND	ug/L	9.8	1.8	1	09/22/22 19:22	09/23/22 17:26	120-12-7	
Benzdine	ND	ug/L	49.0	5.9	1	09/22/22 19:22	09/23/22 17:26	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.8	1.7	1	09/22/22 19:22	09/23/22 17:26	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.8	2.3	1	09/22/22 19:22	09/23/22 17:26	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.8	2.5	1	09/22/22 19:22	09/23/22 17:26	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.8	2.2	1	09/22/22 19:22	09/23/22 17:26	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.8	2.5	1	09/22/22 19:22	09/23/22 17:26	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.8	2.9	1	09/22/22 19:22	09/23/22 17:26	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.8	3.6	1	09/22/22 19:22	09/23/22 17:26	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.6	2.9	1	09/22/22 19:22	09/23/22 17:26	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.8	3.0	1	09/22/22 19:22	09/23/22 17:26	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.8	2.1	1	09/22/22 19:22	09/23/22 17:26	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.8	3.6	1	09/22/22 19:22	09/23/22 17:26	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.8	2.6	1	09/22/22 19:22	09/23/22 17:26	91-58-7	
2-Chlorophenol	ND	ug/L	9.8	2.2	1	09/22/22 19:22	09/23/22 17:26	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.8	2.6	1	09/22/22 19:22	09/23/22 17:26	7005-72-3	
Chrysene	ND	ug/L	9.8	1.9	1	09/22/22 19:22	09/23/22 17:26	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.8	2.5	1	09/22/22 19:22	09/23/22 17:26	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.8	2.7	1	09/22/22 19:22	09/23/22 17:26	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.8	2.8	1	09/22/22 19:22	09/23/22 17:26	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.8	2.9	1	09/22/22 19:22	09/23/22 17:26	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.6	3.1	1	09/22/22 19:22	09/23/22 17:26	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.8	2.3	1	09/22/22 19:22	09/23/22 17:26	120-83-2	
Diethylphthalate	ND	ug/L	9.8	3.1	1	09/22/22 19:22	09/23/22 17:26	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.8	2.2	1	09/22/22 19:22	09/23/22 17:26	105-67-9	
Dimethylphthalate	ND	ug/L	9.8	2.3	1	09/22/22 19:22	09/23/22 17:26	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.8	3.3	1	09/22/22 19:22	09/23/22 17:26	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	49.0	8.0	1	09/22/22 19:22	09/23/22 17:26	534-52-1	
2,4-Dinitrophenol	ND	ug/L	49.0	5.1	1	09/22/22 19:22	09/23/22 17:26	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.8	2.3	1	09/22/22 19:22	09/23/22 17:26	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.8	2.8	1	09/22/22 19:22	09/23/22 17:26	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.8	7.2	1	09/22/22 19:22	09/23/22 17:26	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.8	2.0	1	09/22/22 19:22	09/23/22 17:26	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.9	4.1	1	09/22/22 19:22	09/23/22 17:26	117-81-7	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-EFF-19SEP2022 Lab ID: 50326308003 Collected: 09/19/22 15:35 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	9.8	1.8	1	09/22/22 19:22	09/23/22 17:26	206-44-0	
Fluorene	ND	ug/L	9.8	1.9	1	09/22/22 19:22	09/23/22 17:26	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.8	3.8	1	09/22/22 19:22	09/23/22 17:26	87-68-3	
Hexachlorobenzene	ND	ug/L	9.8	3.3	1	09/22/22 19:22	09/23/22 17:26	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.6	4.4	1	09/22/22 19:22	09/23/22 17:26	77-47-4	N2
Hexachloroethane	ND	ug/L	9.8	3.3	1	09/22/22 19:22	09/23/22 17:26	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.8	2.6	1	09/22/22 19:22	09/23/22 17:26	193-39-5	
Isophorone	ND	ug/L	9.8	1.9	1	09/22/22 19:22	09/23/22 17:26	78-59-1	
Naphthalene	ND	ug/L	9.8	2.3	1	09/22/22 19:22	09/23/22 17:26	91-20-3	
Nitrobenzene	ND	ug/L	9.8	3.1	1	09/22/22 19:22	09/23/22 17:26	98-95-3	
2-Nitrophenol	ND	ug/L	9.8	4.1	1	09/22/22 19:22	09/23/22 17:26	88-75-5	
4-Nitrophenol	ND	ug/L	49.0	5.0	1	09/22/22 19:22	09/23/22 17:26	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.6	3.4	1	09/22/22 19:22	09/23/22 17:26	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.8	2.9	1	09/22/22 19:22	09/23/22 17:26	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.8	2.1	1	09/22/22 19:22	09/23/22 17:26	86-30-6	
Pentachlorophenol	ND	ug/L	49.0	6.6	1	09/22/22 19:22	09/23/22 17:26	87-86-5	
Phenanthrene	ND	ug/L	9.8	1.9	1	09/22/22 19:22	09/23/22 17:26	85-01-8	
Phenol	ND	ug/L	9.8	1.2	1	09/22/22 19:22	09/23/22 17:26	108-95-2	
Pyrene	ND	ug/L	9.8	2.0	1	09/22/22 19:22	09/23/22 17:26	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.8	3.6	1	09/22/22 19:22	09/23/22 17:26	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.8	2.7	1	09/22/22 19:22	09/23/22 17:26	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	41	%	9-74		1	09/22/22 19:22	09/23/22 17:26	367-12-4	
Phenol-d5 (S)	31	%	8-424		1	09/22/22 19:22	09/23/22 17:26	4165-62-2	
Nitrobenzene-d5 (S)	80	%	15-314		1	09/22/22 19:22	09/23/22 17:26	4165-60-0	
2-Fluorobiphenyl (S)	79	%	32-92		1	09/22/22 19:22	09/23/22 17:26	321-60-8	
2,4,6-Tribromophenol (S)	64	%	27-125		1	09/22/22 19:22	09/23/22 17:26	118-79-6	
p-Terphenyl-d14 (S)	88	%	8-146		1	09/22/22 19:22	09/23/22 17:26	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	3.3	1		09/21/22 14:44	107-02-8	L1
Acrylonitrile	ND	ug/L	100	0.50	1		09/21/22 14:44	107-13-1	
Benzene	ND	ug/L	5.0	0.11	1		09/21/22 14:44	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.11	1		09/21/22 14:44	75-27-4	
Bromoform	ND	ug/L	5.0	0.036	1		09/21/22 14:44	75-25-2	
Bromomethane	ND	ug/L	5.0	0.19	1		09/21/22 14:44	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.065	1		09/21/22 14:44	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.098	1		09/21/22 14:44	108-90-7	
Chloroethane	ND	ug/L	5.0	0.17	1		09/21/22 14:44	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	0.37	1		09/21/22 14:44	110-75-8	
Chloroform	ND	ug/L	4.8	0.15	1		09/21/22 14:44	67-66-3	
Chloromethane	ND	ug/L	5.0	0.16	1		09/21/22 14:44	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.041	1		09/21/22 14:44	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.10	1		09/21/22 14:44	75-34-3	

**REPORT OF LABORATORY ANALYSIS**

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-EFF-19SEP2022 Lab ID: 50326308003 Collected: 09/19/22 15:35 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.12	1		09/21/22 14:44	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.13	1		09/21/22 14:44	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.13	1		09/21/22 14:44	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.12	1		09/21/22 14:44	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 14:44	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 14:44	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.082	1		09/21/22 14:44	100-41-4	
Methylene Chloride	ND	ug/L	5.0	1.6	1		09/21/22 14:44	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.090	1		09/21/22 14:44	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.16	1		09/21/22 14:44	127-18-4	
Toluene	ND	ug/L	5.0	0.11	1		09/21/22 14:44	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.097	1		09/21/22 14:44	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.11	1		09/21/22 14:44	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.12	1		09/21/22 14:44	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.11	1		09/21/22 14:44	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	96	%	91-114		1		09/21/22 14:44	1868-53-7	
4-Bromofluorobenzene (S)	98	%	85-120		1		09/21/22 14:44	460-00-4	
Toluene-d8 (S)	101	%	85-117		1		09/21/22 14:44	2037-26-5	

### 335.4 Cyanide, Total

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	<b>0.0032J</b>	mg/L	0.0050	0.0018	1	09/26/22 11:46	09/27/22 17:39	57-12-5	
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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-DNS-19SEP2022 Lab ID: 50326308004 Collected: 09/19/22 12:10 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>608.3 PCB</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
PCB-1016 (Aroclor 1016)	ND	ug/L	0.10	0.059	1	09/22/22 23:01	09/26/22 21:43	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.10	0.059	1	09/22/22 23:01	09/26/22 21:43	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.10	0.059	1	09/22/22 23:01	09/26/22 21:43	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.10	0.059	1	09/22/22 23:01	09/26/22 21:43	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.10	0.059	1	09/22/22 23:01	09/26/22 21:43	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.10	0.059	1	09/22/22 23:01	09/26/22 21:43	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.10	0.052	1	09/22/22 23:01	09/26/22 21:43	11096-82-5	
<b>Surrogates</b>									
Tetrachloro-m-xylene (S)	48	%	1-123		1	09/22/22 23:01	09/26/22 21:43	877-09-8	
<b>608.3 Pesticides</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
Aldrin	ND	ug/L	0.051	0.0091	1	09/22/22 23:01	10/10/22 16:00	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.051	0.011	1	09/22/22 23:01	10/10/22 16:00	319-84-6	
beta-BHC	ND	ug/L	0.051	0.014	1	09/22/22 23:01	10/10/22 16:00	319-85-7	
delta-BHC	ND	ug/L	0.051	0.013	1	09/22/22 23:01	10/10/22 16:00	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.051	0.012	1	09/22/22 23:01	10/10/22 16:00	58-89-9	
Chlordane (Technical)	ND	ug/L	0.51	0.27	1	09/22/22 23:01	10/10/22 16:00	57-74-9	
4,4'-DDD	ND	ug/L	0.10	0.024	1	09/22/22 23:01	10/10/22 16:00	72-54-8	
4,4'-DDE	ND	ug/L	0.10	0.018	1	09/22/22 23:01	10/10/22 16:00	72-55-9	
4,4'-DDT	ND	ug/L	0.10	0.036	1	09/22/22 23:01	10/10/22 16:00	50-29-3	
Dieldrin	ND	ug/L	0.10	0.022	1	09/22/22 23:01	10/10/22 16:00	60-57-1	
Endosulfan I	ND	ug/L	0.051	0.012	1	09/22/22 23:01	10/10/22 16:00	959-98-8	
Endosulfan II	ND	ug/L	0.10	0.025	1	09/22/22 23:01	10/10/22 16:00	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.10	0.021	1	09/22/22 23:01	10/10/22 16:00	1031-07-8	
Endrin	ND	ug/L	0.10	0.027	1	09/22/22 23:01	10/10/22 16:00	72-20-8	
Endrin aldehyde	ND	ug/L	0.10	0.025	1	09/22/22 23:01	10/10/22 16:00	7421-93-4	
Heptachlor	ND	ug/L	0.051	0.010	1	09/22/22 23:01	10/10/22 16:00	76-44-8	H7,L2
Heptachlor epoxide	ND	ug/L	0.051	0.011	1	09/22/22 23:01	10/10/22 16:00	1024-57-3	
Toxaphene	ND	ug/L	1.0	0.36	1	09/22/22 23:01	10/10/22 16:00	8001-35-2	
<b>Surrogates</b>									
Decachlorobiphenyl (S)	38	%	1-140		1	09/22/22 23:01	10/10/22 16:00	2051-24-3	
<b>200.8 Metals, Total ICMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Antimony	0.00088J	mg/L	0.0010	0.00013	1	09/28/22 08:00	10/03/22 14:16	7440-36-0	
Arsenic	0.0036	mg/L	0.0010	0.00011	1	09/28/22 08:00	10/03/22 14:16	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	09/28/22 08:00	10/03/22 14:16	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	09/28/22 08:00	10/03/22 14:16	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	09/28/22 08:00	10/03/22 14:16	7440-47-3	
Copper	0.0058	mg/L	0.0010	0.00037	1	09/28/22 08:00	10/03/22 14:16	7440-50-8	
Lead	0.00016J	mg/L	0.0010	0.000080	1	09/28/22 08:00	10/03/22 14:16	7439-92-1	
Nickel	0.0039	mg/L	0.00050	0.00039	1	09/28/22 08:00	10/03/22 14:16	7440-02-0	
Selenium	0.055	mg/L	0.0010	0.00035	1	09/28/22 08:00	10/03/22 14:16	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	09/28/22 08:00	10/03/22 14:16	7440-22-4	

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-DNS-19SEP2022 Lab ID: 50326308004 Collected: 09/19/22 12:10 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	0.000093J	mg/L	0.0010	0.000073	1	09/28/22 08:00	10/03/22 14:16	7440-28-0	
Zinc	0.0081	mg/L	0.0030	0.0010	1	09/28/22 08:00	10/03/22 14:16	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/03/22 09:26	10/03/22 13:09	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	10.0	1.8	1	09/23/22 14:33	09/27/22 19:51	83-32-9	
Acenaphthylene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 19:51	208-96-8	
Anthracene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 19:51	120-12-7	
Benzdine	ND	ug/L	50.0	6.0	1	09/23/22 14:33	09/27/22 19:51	92-87-5	
Benzo(a)anthracene	ND	ug/L	10.0	1.7	1	09/23/22 14:33	09/27/22 19:51	56-55-3	
Benzo(a)pyrene	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 19:51	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 19:51	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	10.0	2.2	1	09/23/22 14:33	09/27/22 19:51	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 19:51	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 19:51	101-55-3	
Butylbenzylphthalate	ND	ug/L	10.0	3.7	1	09/23/22 14:33	09/27/22 19:51	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	20.0	3.0	1	09/23/22 14:33	09/27/22 19:51	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	10.0	3.1	1	09/23/22 14:33	09/27/22 19:51	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	10.0	2.2	1	09/23/22 14:33	09/27/22 19:51	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	10.0	3.6	1	09/23/22 14:33	09/27/22 19:51	108-60-1	
2-Chloronaphthalene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 19:51	91-58-7	
2-Chlorophenol	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 19:51	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	10.0	2.7	1	09/23/22 14:33	09/27/22 19:51	7005-72-3	
Chrysene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 19:51	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 19:51	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	10.0	2.7	1	09/23/22 14:33	09/27/22 19:51	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 19:51	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 19:51	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	20.0	3.1	1	09/23/22 14:33	09/27/22 19:51	91-94-1	
2,4-Dichlorophenol	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 19:51	120-83-2	
Diethylphthalate	ND	ug/L	10.0	3.2	1	09/23/22 14:33	09/27/22 19:51	84-66-2	
2,4-Dimethylphenol	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 19:51	105-67-9	
Dimethylphthalate	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 19:51	131-11-3	
Di-n-butylphthalate	ND	ug/L	10.0	3.3	1	09/23/22 14:33	09/27/22 19:51	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	50.0	8.2	1	09/23/22 14:33	09/27/22 19:51	534-52-1	
2,4-Dinitrophenol	ND	ug/L	50.0	5.2	1	09/23/22 14:33	09/27/22 19:51	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	10.0	2.4	1	09/23/22 14:33	09/27/22 19:51	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 19:51	606-20-2	
Di-n-octylphthalate	ND	ug/L	10.0	7.3	1	09/23/22 14:33	09/27/22 19:51	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	10.0	2.1	1	09/23/22 14:33	09/27/22 19:51	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	5.0	4.2	1	09/23/22 14:33	09/27/22 19:51	117-81-7	

### REPORT OF LABORATORY ANALYSIS

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 20-DNS-19SEP2022 Lab ID: 50326308004 Collected: 09/19/22 12:10 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	10.0	1.8	1	09/23/22 14:33	09/27/22 19:51	206-44-0	
Fluorene	ND	ug/L	10.0	2.0	1	09/23/22 14:33	09/27/22 19:51	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	10.0	3.8	1	09/23/22 14:33	09/27/22 19:51	87-68-3	
Hexachlorobenzene	ND	ug/L	10.0	3.3	1	09/23/22 14:33	09/27/22 19:51	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	20.0	4.5	1	09/23/22 14:33	09/27/22 19:51	77-47-4	N2
Hexachloroethane	ND	ug/L	10.0	3.4	1	09/23/22 14:33	09/27/22 19:51	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 19:51	193-39-5	
Isophorone	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 19:51	78-59-1	
Naphthalene	ND	ug/L	10.0	2.4	1	09/23/22 14:33	09/27/22 19:51	91-20-3	
Nitrobenzene	ND	ug/L	10.0	3.1	1	09/23/22 14:33	09/27/22 19:51	98-95-3	
2-Nitrophenol	ND	ug/L	10.0	4.2	1	09/23/22 14:33	09/27/22 19:51	88-75-5	
4-Nitrophenol	ND	ug/L	50.0	5.1	1	09/23/22 14:33	09/27/22 19:51	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	20.0	3.5	1	09/23/22 14:33	09/27/22 19:51	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	10.0	3.0	1	09/23/22 14:33	09/27/22 19:51	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	10.0	2.2	1	09/23/22 14:33	09/27/22 19:51	86-30-6	
Pentachlorophenol	ND	ug/L	50.0	6.7	1	09/23/22 14:33	09/27/22 19:51	87-86-5	
Phenanthrene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 19:51	85-01-8	
Phenol	ND	ug/L	10.0	1.2	1	09/23/22 14:33	09/27/22 19:51	108-95-2	
Pyrene	ND	ug/L	10.0	2.0	1	09/23/22 14:33	09/27/22 19:51	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	10.0	3.7	1	09/23/22 14:33	09/27/22 19:51	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	10.0	2.8	1	09/23/22 14:33	09/27/22 19:51	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	39	%	9-74		1	09/23/22 14:33	09/27/22 19:51	367-12-4	
Phenol-d5 (S)	26	%	8-424		1	09/23/22 14:33	09/27/22 19:51	4165-62-2	
Nitrobenzene-d5 (S)	72	%	15-314		1	09/23/22 14:33	09/27/22 19:51	4165-60-0	
2-Fluorobiphenyl (S)	68	%	32-92		1	09/23/22 14:33	09/27/22 19:51	321-60-8	
2,4,6-Tribromophenol (S)	80	%	27-125		1	09/23/22 14:33	09/27/22 19:51	118-79-6	
p-Terphenyl-d14 (S)	85	%	8-146		1	09/23/22 14:33	09/27/22 19:51	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	3.3	1		09/21/22 15:14	107-02-8	L1
Acrylonitrile	ND	ug/L	100	0.50	1		09/21/22 15:14	107-13-1	
Benzene	ND	ug/L	5.0	0.11	1		09/21/22 15:14	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.11	1		09/21/22 15:14	75-27-4	
Bromoform	ND	ug/L	5.0	0.036	1		09/21/22 15:14	75-25-2	
Bromomethane	ND	ug/L	5.0	0.19	1		09/21/22 15:14	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.065	1		09/21/22 15:14	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.098	1		09/21/22 15:14	108-90-7	
Chloroethane	ND	ug/L	5.0	0.17	1		09/21/22 15:14	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	0.37	1		09/21/22 15:14	110-75-8	
Chloroform	ND	ug/L	4.8	0.15	1		09/21/22 15:14	67-66-3	
Chloromethane	ND	ug/L	5.0	0.16	1		09/21/22 15:14	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.041	1		09/21/22 15:14	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.10	1		09/21/22 15:14	75-34-3	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

**Sample: 20-DNS-19SEP2022**      **Lab ID: 50326308004**      Collected: 09/19/22 12:10      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.12	1		09/21/22 15:14	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.13	1		09/21/22 15:14	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.13	1		09/21/22 15:14	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.12	1		09/21/22 15:14	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 15:14	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 15:14	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.082	1		09/21/22 15:14	100-41-4	
Methylene Chloride	ND	ug/L	5.0	1.6	1		09/21/22 15:14	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.090	1		09/21/22 15:14	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.16	1		09/21/22 15:14	127-18-4	
Toluene	ND	ug/L	5.0	0.11	1		09/21/22 15:14	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.097	1		09/21/22 15:14	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.11	1		09/21/22 15:14	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.12	1		09/21/22 15:14	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.11	1		09/21/22 15:14	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	96	%	91-114		1		09/21/22 15:14	1868-53-7	
4-Bromofluorobenzene (S)	100	%	85-120		1		09/21/22 15:14	460-00-4	
Toluene-d8 (S)	102	%	85-117		1		09/21/22 15:14	2037-26-5	
<b>335.4 Cyanide, Total</b>									
Analytical Method: EPA 335.4      Preparation Method: EPA 335.4									
Pace Analytical Services - Indianapolis									
Cyanide	<b>0.0022J</b>	mg/L	0.0050	0.0018	1	09/26/22 11:46	09/27/22 17:40	57-12-5	

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 30-CON-19SEP2022 Lab ID: 50326308005 Collected: 09/19/22 16:05 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>608.3 PCB</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
PCB-1016 (Aroclor 1016)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:58	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:58	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:58	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:58	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:58	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.10	0.058	1	09/22/22 23:01	09/26/22 21:58	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.10	0.051	1	09/22/22 23:01	09/26/22 21:58	11096-82-5	
<b>Surrogates</b>									
Tetrachloro-m-xylene (S)	57	%	1-123		1	09/22/22 23:01	09/26/22 21:58	877-09-8	
<b>608.3 Pesticides</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
Aldrin	ND	ug/L	0.050	0.0090	1	09/22/22 23:01	10/10/22 16:12	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.050	0.011	1	09/22/22 23:01	10/10/22 16:12	319-84-6	
beta-BHC	ND	ug/L	0.050	0.014	1	09/22/22 23:01	10/10/22 16:12	319-85-7	
delta-BHC	ND	ug/L	0.050	0.013	1	09/22/22 23:01	10/10/22 16:12	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.050	0.012	1	09/22/22 23:01	10/10/22 16:12	58-89-9	
Chlordane (Technical)	ND	ug/L	0.50	0.27	1	09/22/22 23:01	10/10/22 16:12	57-74-9	
4,4'-DDD	ND	ug/L	0.10	0.024	1	09/22/22 23:01	10/10/22 16:12	72-54-8	
4,4'-DDE	ND	ug/L	0.10	0.018	1	09/22/22 23:01	10/10/22 16:12	72-55-9	
4,4'-DDT	ND	ug/L	0.10	0.036	1	09/22/22 23:01	10/10/22 16:12	50-29-3	
Dieldrin	ND	ug/L	0.10	0.022	1	09/22/22 23:01	10/10/22 16:12	60-57-1	
Endosulfan I	ND	ug/L	0.050	0.012	1	09/22/22 23:01	10/10/22 16:12	959-98-8	
Endosulfan II	ND	ug/L	0.10	0.025	1	09/22/22 23:01	10/10/22 16:12	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.10	0.021	1	09/22/22 23:01	10/10/22 16:12	1031-07-8	
Endrin	ND	ug/L	0.10	0.027	1	09/22/22 23:01	10/10/22 16:12	72-20-8	
Endrin aldehyde	ND	ug/L	0.10	0.025	1	09/22/22 23:01	10/10/22 16:12	7421-93-4	
Heptachlor	ND	ug/L	0.050	0.010	1	09/22/22 23:01	10/10/22 16:12	76-44-8	H7,L2
Heptachlor epoxide	ND	ug/L	0.050	0.011	1	09/22/22 23:01	10/10/22 16:12	1024-57-3	
Toxaphene	ND	ug/L	1.0	0.36	1	09/22/22 23:01	10/10/22 16:12	8001-35-2	
<b>Surrogates</b>									
Decachlorobiphenyl (S)	75	%	1-140		1	09/22/22 23:01	10/10/22 16:12	2051-24-3	
<b>200.8 Metals, Total ICMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Antimony	ND	mg/L	0.0010	0.00013	1	09/28/22 08:00	10/03/22 14:25	7440-36-0	
Arsenic	<b>0.00048J</b>	mg/L	0.0010	0.00011	1	09/28/22 08:00	10/03/22 14:25	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	09/28/22 08:00	10/03/22 14:25	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	09/28/22 08:00	10/03/22 14:25	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	09/28/22 08:00	10/03/22 14:25	7440-47-3	
Copper	<b>0.0048</b>	mg/L	0.0010	0.00037	1	09/28/22 08:00	10/03/22 14:25	7440-50-8	
Lead	ND	mg/L	0.0010	0.000080	1	09/28/22 08:00	10/03/22 14:25	7439-92-1	
Nickel	<b>0.0012</b>	mg/L	0.00050	0.00039	1	09/28/22 08:00	10/03/22 14:25	7440-02-0	
Selenium	<b>0.0020</b>	mg/L	0.0010	0.00035	1	09/28/22 08:00	10/03/22 14:25	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	09/28/22 08:00	10/03/22 14:25	7440-22-4	

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 30-CON-19SEP2022 Lab ID: 50326308005 Collected: 09/19/22 16:05 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	ND	mg/L	0.0010	0.000073	1	09/28/22 08:00	10/03/22 14:25	7440-28-0	
Zinc	0.031	mg/L	0.0030	0.0010	1	09/28/22 08:00	10/03/22 14:25	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/03/22 09:26	10/03/22 13:11	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	10.0	1.8	1	09/23/22 14:33	09/27/22 20:06	83-32-9	
Acenaphthylene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:06	208-96-8	
Anthracene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:06	120-12-7	
Benzdine	ND	ug/L	50.0	6.0	1	09/23/22 14:33	09/27/22 20:06	92-87-5	
Benzo(a)anthracene	ND	ug/L	10.0	1.7	1	09/23/22 14:33	09/27/22 20:06	56-55-3	
Benzo(a)pyrene	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:06	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:06	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	10.0	2.2	1	09/23/22 14:33	09/27/22 20:06	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:06	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 20:06	101-55-3	
Butylbenzylphthalate	ND	ug/L	10.0	3.7	1	09/23/22 14:33	09/27/22 20:06	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	20.0	3.0	1	09/23/22 14:33	09/27/22 20:06	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	10.0	3.1	1	09/23/22 14:33	09/27/22 20:06	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	10.0	2.2	1	09/23/22 14:33	09/27/22 20:06	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	10.0	3.6	1	09/23/22 14:33	09/27/22 20:06	108-60-1	
2-Chloronaphthalene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:06	91-58-7	
2-Chlorophenol	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:06	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	10.0	2.7	1	09/23/22 14:33	09/27/22 20:06	7005-72-3	
Chrysene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:06	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:06	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	10.0	2.7	1	09/23/22 14:33	09/27/22 20:06	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 20:06	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 20:06	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	20.0	3.1	1	09/23/22 14:33	09/27/22 20:06	91-94-1	
2,4-Dichlorophenol	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:06	120-83-2	
Diethylphthalate	ND	ug/L	10.0	3.2	1	09/23/22 14:33	09/27/22 20:06	84-66-2	
2,4-Dimethylphenol	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:06	105-67-9	
Dimethylphthalate	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:06	131-11-3	
Di-n-butylphthalate	ND	ug/L	10.0	3.3	1	09/23/22 14:33	09/27/22 20:06	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	50.0	8.2	1	09/23/22 14:33	09/27/22 20:06	534-52-1	
2,4-Dinitrophenol	ND	ug/L	50.0	5.2	1	09/23/22 14:33	09/27/22 20:06	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	10.0	2.4	1	09/23/22 14:33	09/27/22 20:06	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 20:06	606-20-2	
Di-n-octylphthalate	ND	ug/L	10.0	7.3	1	09/23/22 14:33	09/27/22 20:06	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	10.0	2.1	1	09/23/22 14:33	09/27/22 20:06	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	5.0	4.2	1	09/23/22 14:33	09/27/22 20:06	117-81-7	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 30-CON-19SEP2022 Lab ID: 50326308005 Collected: 09/19/22 16:05 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	10.0	1.8	1	09/23/22 14:33	09/27/22 20:06	206-44-0	
Fluorene	ND	ug/L	10.0	2.0	1	09/23/22 14:33	09/27/22 20:06	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	10.0	3.8	1	09/23/22 14:33	09/27/22 20:06	87-68-3	
Hexachlorobenzene	ND	ug/L	10.0	3.3	1	09/23/22 14:33	09/27/22 20:06	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	20.0	4.5	1	09/23/22 14:33	09/27/22 20:06	77-47-4	N2
Hexachloroethane	ND	ug/L	10.0	3.4	1	09/23/22 14:33	09/27/22 20:06	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:06	193-39-5	
Isophorone	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:06	78-59-1	
Naphthalene	ND	ug/L	10.0	2.4	1	09/23/22 14:33	09/27/22 20:06	91-20-3	
Nitrobenzene	ND	ug/L	10.0	3.1	1	09/23/22 14:33	09/27/22 20:06	98-95-3	
2-Nitrophenol	ND	ug/L	10.0	4.2	1	09/23/22 14:33	09/27/22 20:06	88-75-5	
4-Nitrophenol	ND	ug/L	50.0	5.1	1	09/23/22 14:33	09/27/22 20:06	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	20.0	3.5	1	09/23/22 14:33	09/27/22 20:06	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	10.0	3.0	1	09/23/22 14:33	09/27/22 20:06	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	10.0	2.2	1	09/23/22 14:33	09/27/22 20:06	86-30-6	
Pentachlorophenol	ND	ug/L	50.0	6.7	1	09/23/22 14:33	09/27/22 20:06	87-86-5	
Phenanthrene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:06	85-01-8	
Phenol	ND	ug/L	10.0	1.2	1	09/23/22 14:33	09/27/22 20:06	108-95-2	
Pyrene	ND	ug/L	10.0	2.0	1	09/23/22 14:33	09/27/22 20:06	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	10.0	3.7	1	09/23/22 14:33	09/27/22 20:06	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	10.0	2.8	1	09/23/22 14:33	09/27/22 20:06	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	39	%	9-74		1	09/23/22 14:33	09/27/22 20:06	367-12-4	
Phenol-d5 (S)	25	%	8-424		1	09/23/22 14:33	09/27/22 20:06	4165-62-2	
Nitrobenzene-d5 (S)	66	%	15-314		1	09/23/22 14:33	09/27/22 20:06	4165-60-0	
2-Fluorobiphenyl (S)	64	%	32-92		1	09/23/22 14:33	09/27/22 20:06	321-60-8	
2,4,6-Tribromophenol (S)	73	%	27-125		1	09/23/22 14:33	09/27/22 20:06	118-79-6	
p-Terphenyl-d14 (S)	84	%	8-146		1	09/23/22 14:33	09/27/22 20:06	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	3.3	1		09/21/22 15:43	107-02-8	L1
Acrylonitrile	ND	ug/L	100	0.50	1		09/21/22 15:43	107-13-1	
Benzene	ND	ug/L	5.0	0.11	1		09/21/22 15:43	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.11	1		09/21/22 15:43	75-27-4	
Bromoform	ND	ug/L	5.0	0.036	1		09/21/22 15:43	75-25-2	
Bromomethane	ND	ug/L	5.0	0.19	1		09/21/22 15:43	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.065	1		09/21/22 15:43	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.098	1		09/21/22 15:43	108-90-7	
Chloroethane	ND	ug/L	5.0	0.17	1		09/21/22 15:43	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	0.37	1		09/21/22 15:43	110-75-8	
Chloroform	ND	ug/L	4.8	0.15	1		09/21/22 15:43	67-66-3	
Chloromethane	ND	ug/L	5.0	0.16	1		09/21/22 15:43	74-87-3	
Dibromochloromethane	0.56J	ug/L	5.0	0.041	1		09/21/22 15:43	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.10	1		09/21/22 15:43	75-34-3	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

**Sample: 30-CON-19SEP2022**      **Lab ID: 50326308005**      Collected: 09/19/22 16:05      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.12	1		09/21/22 15:43	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.13	1		09/21/22 15:43	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.13	1		09/21/22 15:43	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.12	1		09/21/22 15:43	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 15:43	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 15:43	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.082	1		09/21/22 15:43	100-41-4	
Methylene Chloride	ND	ug/L	5.0	1.6	1		09/21/22 15:43	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.090	1		09/21/22 15:43	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.16	1		09/21/22 15:43	127-18-4	
Toluene	ND	ug/L	5.0	0.11	1		09/21/22 15:43	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.097	1		09/21/22 15:43	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.11	1		09/21/22 15:43	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.12	1		09/21/22 15:43	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.11	1		09/21/22 15:43	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	96	%	91-114		1		09/21/22 15:43	1868-53-7	
4-Bromofluorobenzene (S)	100	%	85-120		1		09/21/22 15:43	460-00-4	
Toluene-d8 (S)	102	%	85-117		1		09/21/22 15:43	2037-26-5	

**335.4 Cyanide, Total**

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	ND	mg/L	0.0050	0.0018	1	09/26/22 11:46	09/27/22 17:42	57-12-5	
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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 30-UPS-19SEP2022 Lab ID: 50326308006 Collected: 09/19/22 13:30 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>608.3 PCB</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
PCB-1016 (Aroclor 1016)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 22:13	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 22:13	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 22:13	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 22:13	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 22:13	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.098	0.057	1	09/22/22 23:01	09/26/22 22:13	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.098	0.050	1	09/22/22 23:01	09/26/22 22:13	11096-82-5	
<b>Surrogates</b>									
Tetrachloro-m-xylene (S)	48	%	1-123		1	09/22/22 23:01	09/26/22 22:13	877-09-8	
<b>608.3 Pesticides</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
Aldrin	ND	ug/L	0.049	0.0088	1	09/22/22 23:01	10/10/22 16:25	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.049	0.011	1	09/22/22 23:01	10/10/22 16:25	319-84-6	
beta-BHC	ND	ug/L	0.049	0.014	1	09/22/22 23:01	10/10/22 16:25	319-85-7	
delta-BHC	ND	ug/L	0.049	0.013	1	09/22/22 23:01	10/10/22 16:25	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.049	0.012	1	09/22/22 23:01	10/10/22 16:25	58-89-9	
Chlordane (Technical)	ND	ug/L	0.49	0.26	1	09/22/22 23:01	10/10/22 16:25	57-74-9	
4,4'-DDD	ND	ug/L	0.098	0.024	1	09/22/22 23:01	10/10/22 16:25	72-54-8	
4,4'-DDE	ND	ug/L	0.098	0.018	1	09/22/22 23:01	10/10/22 16:25	72-55-9	
4,4'-DDT	ND	ug/L	0.098	0.035	1	09/22/22 23:01	10/10/22 16:25	50-29-3	
Dieldrin	ND	ug/L	0.098	0.022	1	09/22/22 23:01	10/10/22 16:25	60-57-1	
Endosulfan I	ND	ug/L	0.049	0.012	1	09/22/22 23:01	10/10/22 16:25	959-98-8	
Endosulfan II	ND	ug/L	0.098	0.025	1	09/22/22 23:01	10/10/22 16:25	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.098	0.021	1	09/22/22 23:01	10/10/22 16:25	1031-07-8	
Endrin	ND	ug/L	0.098	0.026	1	09/22/22 23:01	10/10/22 16:25	72-20-8	
Endrin aldehyde	ND	ug/L	0.098	0.025	1	09/22/22 23:01	10/10/22 16:25	7421-93-4	
Heptachlor	ND	ug/L	0.049	0.0098	1	09/22/22 23:01	10/10/22 16:25	76-44-8	H7,L2
Heptachlor epoxide	ND	ug/L	0.049	0.011	1	09/22/22 23:01	10/10/22 16:25	1024-57-3	
Toxaphene	ND	ug/L	0.98	0.35	1	09/22/22 23:01	10/10/22 16:25	8001-35-2	
<b>Surrogates</b>									
Decachlorobiphenyl (S)	56	%	1-140		1	09/22/22 23:01	10/10/22 16:25	2051-24-3	
<b>200.8 Metals, Total ICMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Antimony	0.00033J	mg/L	0.0010	0.00013	1	09/28/22 08:00	10/03/22 14:37	7440-36-0	
Arsenic	0.0014	mg/L	0.0010	0.00011	1	09/28/22 08:00	10/03/22 14:37	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	09/28/22 08:00	10/03/22 14:37	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	09/28/22 08:00	10/03/22 14:37	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	09/28/22 08:00	10/03/22 14:37	7440-47-3	
Copper	0.0020	mg/L	0.0010	0.00037	1	09/28/22 08:00	10/03/22 14:37	7440-50-8	
Lead	0.00011J	mg/L	0.0010	0.000080	1	09/28/22 08:00	10/03/22 14:37	7439-92-1	
Nickel	0.0032	mg/L	0.00050	0.00039	1	09/28/22 08:00	10/03/22 14:37	7440-02-0	
Selenium	0.00074J	mg/L	0.0010	0.00035	1	09/28/22 08:00	10/03/22 14:37	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	09/28/22 08:00	10/03/22 14:37	7440-22-4	

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

**Sample: 30-UPS-19SEP2022**      **Lab ID: 50326308006**      Collected: 09/19/22 13:30      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	ND	mg/L	0.0010	0.000073	1	09/28/22 08:00	10/03/22 14:37	7440-28-0	
Zinc	<b>0.022</b>	mg/L	0.0030	0.0010	1	09/28/22 08:00	10/03/22 14:37	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/03/22 09:26	10/03/22 13:14	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.6	1.7	1	09/23/22 14:33	09/27/22 20:22	83-32-9	
Acenaphthylene	ND	ug/L	9.6	1.8	1	09/23/22 14:33	09/27/22 20:22	208-96-8	
Anthracene	ND	ug/L	9.6	1.8	1	09/23/22 14:33	09/27/22 20:22	120-12-7	
Benzdine	ND	ug/L	48.1	5.8	1	09/23/22 14:33	09/27/22 20:22	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.6	1.6	1	09/23/22 14:33	09/27/22 20:22	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.6	2.2	1	09/23/22 14:33	09/27/22 20:22	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.6	2.5	1	09/23/22 14:33	09/27/22 20:22	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.6	2.1	1	09/23/22 14:33	09/27/22 20:22	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.6	2.5	1	09/23/22 14:33	09/27/22 20:22	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.6	2.8	1	09/23/22 14:33	09/27/22 20:22	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.6	3.6	1	09/23/22 14:33	09/27/22 20:22	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.2	2.9	1	09/23/22 14:33	09/27/22 20:22	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.6	3.0	1	09/23/22 14:33	09/27/22 20:22	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.6	2.1	1	09/23/22 14:33	09/27/22 20:22	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.6	3.5	1	09/23/22 14:33	09/27/22 20:22	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.6	2.5	1	09/23/22 14:33	09/27/22 20:22	91-58-7	
2-Chlorophenol	ND	ug/L	9.6	2.2	1	09/23/22 14:33	09/27/22 20:22	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.6	2.6	1	09/23/22 14:33	09/27/22 20:22	7005-72-3	
Chrysene	ND	ug/L	9.6	1.8	1	09/23/22 14:33	09/27/22 20:22	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.6	2.5	1	09/23/22 14:33	09/27/22 20:22	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.6	2.6	1	09/23/22 14:33	09/27/22 20:22	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.6	2.7	1	09/23/22 14:33	09/27/22 20:22	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.6	2.8	1	09/23/22 14:33	09/27/22 20:22	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.2	3.0	1	09/23/22 14:33	09/27/22 20:22	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.6	2.2	1	09/23/22 14:33	09/27/22 20:22	120-83-2	
Diethylphthalate	ND	ug/L	9.6	3.0	1	09/23/22 14:33	09/27/22 20:22	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.6	2.2	1	09/23/22 14:33	09/27/22 20:22	105-67-9	
Dimethylphthalate	ND	ug/L	9.6	2.2	1	09/23/22 14:33	09/27/22 20:22	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.6	3.2	1	09/23/22 14:33	09/27/22 20:22	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	48.1	7.8	1	09/23/22 14:33	09/27/22 20:22	534-52-1	
2,4-Dinitrophenol	ND	ug/L	48.1	5.0	1	09/23/22 14:33	09/27/22 20:22	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.6	2.3	1	09/23/22 14:33	09/27/22 20:22	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.6	2.7	1	09/23/22 14:33	09/27/22 20:22	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.6	7.1	1	09/23/22 14:33	09/27/22 20:22	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.6	2.0	1	09/23/22 14:33	09/27/22 20:22	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.8	4.0	1	09/23/22 14:33	09/27/22 20:22	117-81-7	

**REPORT OF LABORATORY ANALYSIS**

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 30-UPS-19SEP2022 Lab ID: 50326308006 Collected: 09/19/22 13:30 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	9.6	1.8	1	09/23/22 14:33	09/27/22 20:22	206-44-0	
Fluorene	ND	ug/L	9.6	1.9	1	09/23/22 14:33	09/27/22 20:22	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.6	3.7	1	09/23/22 14:33	09/27/22 20:22	87-68-3	
Hexachlorobenzene	ND	ug/L	9.6	3.2	1	09/23/22 14:33	09/27/22 20:22	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.2	4.4	1	09/23/22 14:33	09/27/22 20:22	77-47-4	N2
Hexachloroethane	ND	ug/L	9.6	3.3	1	09/23/22 14:33	09/27/22 20:22	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.6	2.5	1	09/23/22 14:33	09/27/22 20:22	193-39-5	
Isophorone	ND	ug/L	9.6	1.9	1	09/23/22 14:33	09/27/22 20:22	78-59-1	
Naphthalene	ND	ug/L	9.6	2.3	1	09/23/22 14:33	09/27/22 20:22	91-20-3	
Nitrobenzene	ND	ug/L	9.6	3.0	1	09/23/22 14:33	09/27/22 20:22	98-95-3	
2-Nitrophenol	ND	ug/L	9.6	4.0	1	09/23/22 14:33	09/27/22 20:22	88-75-5	
4-Nitrophenol	ND	ug/L	48.1	4.9	1	09/23/22 14:33	09/27/22 20:22	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.2	3.4	1	09/23/22 14:33	09/27/22 20:22	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.6	2.8	1	09/23/22 14:33	09/27/22 20:22	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.6	2.1	1	09/23/22 14:33	09/27/22 20:22	86-30-6	
Pentachlorophenol	ND	ug/L	48.1	6.5	1	09/23/22 14:33	09/27/22 20:22	87-86-5	
Phenanthrene	ND	ug/L	9.6	1.8	1	09/23/22 14:33	09/27/22 20:22	85-01-8	
Phenol	ND	ug/L	9.6	1.2	1	09/23/22 14:33	09/27/22 20:22	108-95-2	
Pyrene	ND	ug/L	9.6	1.9	1	09/23/22 14:33	09/27/22 20:22	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.6	3.5	1	09/23/22 14:33	09/27/22 20:22	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.6	2.6	1	09/23/22 14:33	09/27/22 20:22	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	39	%	9-74		1	09/23/22 14:33	09/27/22 20:22	367-12-4	
Phenol-d5 (S)	26	%	8-424		1	09/23/22 14:33	09/27/22 20:22	4165-62-2	
Nitrobenzene-d5 (S)	70	%	15-314		1	09/23/22 14:33	09/27/22 20:22	4165-60-0	
2-Fluorobiphenyl (S)	65	%	32-92		1	09/23/22 14:33	09/27/22 20:22	321-60-8	
2,4,6-Tribromophenol (S)	83	%	27-125		1	09/23/22 14:33	09/27/22 20:22	118-79-6	
p-Terphenyl-d14 (S)	92	%	8-146		1	09/23/22 14:33	09/27/22 20:22	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	3.3	1		09/21/22 16:12	107-02-8	L1
Acrylonitrile	ND	ug/L	100	0.50	1		09/21/22 16:12	107-13-1	
Benzene	ND	ug/L	5.0	0.11	1		09/21/22 16:12	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.11	1		09/21/22 16:12	75-27-4	
Bromoform	ND	ug/L	5.0	0.036	1		09/21/22 16:12	75-25-2	
Bromomethane	ND	ug/L	5.0	0.19	1		09/21/22 16:12	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.065	1		09/21/22 16:12	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.098	1		09/21/22 16:12	108-90-7	
Chloroethane	ND	ug/L	5.0	0.17	1		09/21/22 16:12	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	0.37	1		09/21/22 16:12	110-75-8	
Chloroform	ND	ug/L	4.8	0.15	1		09/21/22 16:12	67-66-3	
Chloromethane	ND	ug/L	5.0	0.16	1		09/21/22 16:12	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.041	1		09/21/22 16:12	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.10	1		09/21/22 16:12	75-34-3	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

**Sample: 30-UPS-19SEP2022**      **Lab ID: 50326308006**      Collected: 09/19/22 13:30      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.12	1		09/21/22 16:12	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.13	1		09/21/22 16:12	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.13	1		09/21/22 16:12	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.12	1		09/21/22 16:12	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 16:12	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 16:12	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.082	1		09/21/22 16:12	100-41-4	
Methylene Chloride	ND	ug/L	5.0	1.6	1		09/21/22 16:12	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.090	1		09/21/22 16:12	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.16	1		09/21/22 16:12	127-18-4	
Toluene	ND	ug/L	5.0	0.11	1		09/21/22 16:12	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.097	1		09/21/22 16:12	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.11	1		09/21/22 16:12	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.12	1		09/21/22 16:12	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.11	1		09/21/22 16:12	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	96	%	91-114		1		09/21/22 16:12	1868-53-7	
4-Bromofluorobenzene (S)	99	%	85-120		1		09/21/22 16:12	460-00-4	
Toluene-d8 (S)	102	%	85-117		1		09/21/22 16:12	2037-26-5	

**335.4 Cyanide, Total**

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	<b>0.0020J</b>	mg/L	0.0050	0.0018	1	09/26/22 11:46	09/27/22 17:42	57-12-5	
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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

Sample: 30-EFF-19SEP2022 Lab ID: 50326308007 Collected: 09/19/22 16:20 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>608.3 PCB</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
PCB-1016 (Aroclor 1016)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:44	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:44	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:44	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:44	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:44	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:44	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.10	0.051	1	09/26/22 22:04	09/28/22 20:44	11096-82-5	
<b>Surrogates</b>									
Tetrachloro-m-xylene (S)	60	%	1-123		1	09/26/22 22:04	09/28/22 20:44	877-09-8	
<b>608.3 Pesticides</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
Aldrin	ND	ug/L	0.050	0.0090	1	09/26/22 22:04	10/10/22 14:57	309-00-2	
alpha-BHC	ND	ug/L	0.050	0.011	1	09/26/22 22:04	10/10/22 14:57	319-84-6	
beta-BHC	ND	ug/L	0.050	0.014	1	09/26/22 22:04	10/10/22 14:57	319-85-7	
delta-BHC	ND	ug/L	0.050	0.013	1	09/26/22 22:04	10/10/22 14:57	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.050	0.012	1	09/26/22 22:04	10/10/22 14:57	58-89-9	
Chlordane (Technical)	ND	ug/L	0.50	0.27	1	09/26/22 22:04	10/10/22 14:57	57-74-9	
4,4'-DDD	ND	ug/L	0.10	0.024	1	09/26/22 22:04	10/10/22 14:57	72-54-8	
4,4'-DDE	ND	ug/L	0.10	0.018	1	09/26/22 22:04	10/10/22 14:57	72-55-9	
4,4'-DDT	ND	ug/L	0.10	0.036	1	09/26/22 22:04	10/10/22 14:57	50-29-3	
Dieldrin	ND	ug/L	0.10	0.022	1	09/26/22 22:04	10/10/22 14:57	60-57-1	
Endosulfan I	ND	ug/L	0.050	0.012	1	09/26/22 22:04	10/10/22 14:57	959-98-8	
Endosulfan II	ND	ug/L	0.10	0.025	1	09/26/22 22:04	10/10/22 14:57	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.10	0.021	1	09/26/22 22:04	10/10/22 14:57	1031-07-8	
Endrin	ND	ug/L	0.10	0.027	1	09/26/22 22:04	10/10/22 14:57	72-20-8	
Endrin aldehyde	ND	ug/L	0.10	0.025	1	09/26/22 22:04	10/10/22 14:57	7421-93-4	
Heptachlor	ND	ug/L	0.050	0.010	1	09/26/22 22:04	10/10/22 14:57	76-44-8	
Heptachlor epoxide	ND	ug/L	0.050	0.011	1	09/26/22 22:04	10/10/22 14:57	1024-57-3	
Toxaphene	ND	ug/L	1.0	0.36	1	09/26/22 22:04	10/10/22 14:57	8001-35-2	
<b>Surrogates</b>									
Decachlorobiphenyl (S)	38	%	1-140		1	09/26/22 22:04	10/10/22 14:57	2051-24-3	
<b>200.8 Metals, Total ICMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Antimony	0.0017	mg/L	0.0010	0.00013	1	09/28/22 08:00	10/03/22 14:41	7440-36-0	
Arsenic	0.0045	mg/L	0.0010	0.00011	1	09/28/22 08:00	10/03/22 14:41	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	09/28/22 08:00	10/03/22 14:41	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	09/28/22 08:00	10/03/22 14:41	7440-43-9	
Chromium	0.00071J	mg/L	0.0020	0.00063	1	09/28/22 08:00	10/03/22 14:41	7440-47-3	
Copper	0.0082	mg/L	0.0010	0.00037	1	09/28/22 08:00	10/03/22 14:41	7440-50-8	
Lead	ND	mg/L	0.0010	0.000080	1	09/28/22 08:00	10/03/22 14:41	7439-92-1	
Nickel	0.0051	mg/L	0.00050	0.00039	1	09/28/22 08:00	10/03/22 14:41	7440-02-0	
Selenium	0.11	mg/L	0.0010	0.00035	1	09/28/22 08:00	10/03/22 14:41	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	09/28/22 08:00	10/03/22 14:41	7440-22-4	

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 30-EFF-19SEP2022 Lab ID: 50326308007 Collected: 09/19/22 16:20 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	0.00025J	mg/L	0.0010	0.000073	1	09/28/22 08:00	10/03/22 14:41	7440-28-0	
Zinc	0.016	mg/L	0.0030	0.0010	1	09/28/22 08:00	10/03/22 14:41	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/03/22 09:26	10/03/22 13:16	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.9	1.8	1	09/23/22 14:33	09/27/22 20:38	83-32-9	
Acenaphthylene	ND	ug/L	9.9	1.9	1	09/23/22 14:33	09/27/22 20:38	208-96-8	
Anthracene	ND	ug/L	9.9	1.8	1	09/23/22 14:33	09/27/22 20:38	120-12-7	
Benzdine	ND	ug/L	49.5	5.9	1	09/23/22 14:33	09/27/22 20:38	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.9	1.7	1	09/23/22 14:33	09/27/22 20:38	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.9	2.3	1	09/23/22 14:33	09/27/22 20:38	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.9	2.5	1	09/23/22 14:33	09/27/22 20:38	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.9	2.2	1	09/23/22 14:33	09/27/22 20:38	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.9	2.6	1	09/23/22 14:33	09/27/22 20:38	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.9	2.9	1	09/23/22 14:33	09/27/22 20:38	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.9	3.7	1	09/23/22 14:33	09/27/22 20:38	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.8	3.0	1	09/23/22 14:33	09/27/22 20:38	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.9	3.1	1	09/23/22 14:33	09/27/22 20:38	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.9	2.1	1	09/23/22 14:33	09/27/22 20:38	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.9	3.6	1	09/23/22 14:33	09/27/22 20:38	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.9	2.6	1	09/23/22 14:33	09/27/22 20:38	91-58-7	
2-Chlorophenol	ND	ug/L	9.9	2.3	1	09/23/22 14:33	09/27/22 20:38	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.9	2.7	1	09/23/22 14:33	09/27/22 20:38	7005-72-3	
Chrysene	ND	ug/L	9.9	1.9	1	09/23/22 14:33	09/27/22 20:38	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.9	2.6	1	09/23/22 14:33	09/27/22 20:38	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.9	2.7	1	09/23/22 14:33	09/27/22 20:38	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.9	2.8	1	09/23/22 14:33	09/27/22 20:38	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.9	2.9	1	09/23/22 14:33	09/27/22 20:38	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.8	3.1	1	09/23/22 14:33	09/27/22 20:38	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.9	2.3	1	09/23/22 14:33	09/27/22 20:38	120-83-2	
Diethylphthalate	ND	ug/L	9.9	3.1	1	09/23/22 14:33	09/27/22 20:38	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.9	2.2	1	09/23/22 14:33	09/27/22 20:38	105-67-9	
Dimethylphthalate	ND	ug/L	9.9	2.3	1	09/23/22 14:33	09/27/22 20:38	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.9	3.3	1	09/23/22 14:33	09/27/22 20:38	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	49.5	8.1	1	09/23/22 14:33	09/27/22 20:38	534-52-1	
2,4-Dinitrophenol	ND	ug/L	49.5	5.1	1	09/23/22 14:33	09/27/22 20:38	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.9	2.4	1	09/23/22 14:33	09/27/22 20:38	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.9	2.8	1	09/23/22 14:33	09/27/22 20:38	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.9	7.3	1	09/23/22 14:33	09/27/22 20:38	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.9	2.0	1	09/23/22 14:33	09/27/22 20:38	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	5.0	4.2	1	09/23/22 14:33	09/27/22 20:38	117-81-7	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 30-EFF-19SEP2022 Lab ID: 50326308007 Collected: 09/19/22 16:20 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	9.9	1.8	1	09/23/22 14:33	09/27/22 20:38	206-44-0	
Fluorene	ND	ug/L	9.9	1.9	1	09/23/22 14:33	09/27/22 20:38	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.9	3.8	1	09/23/22 14:33	09/27/22 20:38	87-68-3	
Hexachlorobenzene	ND	ug/L	9.9	3.3	1	09/23/22 14:33	09/27/22 20:38	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.8	4.5	1	09/23/22 14:33	09/27/22 20:38	77-47-4	N2
Hexachloroethane	ND	ug/L	9.9	3.4	1	09/23/22 14:33	09/27/22 20:38	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.9	2.6	1	09/23/22 14:33	09/27/22 20:38	193-39-5	
Isophorone	ND	ug/L	9.9	1.9	1	09/23/22 14:33	09/27/22 20:38	78-59-1	
Naphthalene	ND	ug/L	9.9	2.4	1	09/23/22 14:33	09/27/22 20:38	91-20-3	
Nitrobenzene	ND	ug/L	9.9	3.1	1	09/23/22 14:33	09/27/22 20:38	98-95-3	
2-Nitrophenol	ND	ug/L	9.9	4.2	1	09/23/22 14:33	09/27/22 20:38	88-75-5	
4-Nitrophenol	ND	ug/L	49.5	5.1	1	09/23/22 14:33	09/27/22 20:38	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.8	3.5	1	09/23/22 14:33	09/27/22 20:38	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.9	2.9	1	09/23/22 14:33	09/27/22 20:38	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.9	2.2	1	09/23/22 14:33	09/27/22 20:38	86-30-6	
Pentachlorophenol	ND	ug/L	49.5	6.7	1	09/23/22 14:33	09/27/22 20:38	87-86-5	
Phenanthrene	ND	ug/L	9.9	1.9	1	09/23/22 14:33	09/27/22 20:38	85-01-8	
Phenol	ND	ug/L	9.9	1.2	1	09/23/22 14:33	09/27/22 20:38	108-95-2	
Pyrene	ND	ug/L	9.9	2.0	1	09/23/22 14:33	09/27/22 20:38	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.9	3.6	1	09/23/22 14:33	09/27/22 20:38	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.9	2.7	1	09/23/22 14:33	09/27/22 20:38	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	41	%	9-74		1	09/23/22 14:33	09/27/22 20:38	367-12-4	
Phenol-d5 (S)	29	%	8-424		1	09/23/22 14:33	09/27/22 20:38	4165-62-2	
Nitrobenzene-d5 (S)	72	%	15-314		1	09/23/22 14:33	09/27/22 20:38	4165-60-0	
2-Fluorobiphenyl (S)	69	%	32-92		1	09/23/22 14:33	09/27/22 20:38	321-60-8	
2,4,6-Tribromophenol (S)	84	%	27-125		1	09/23/22 14:33	09/27/22 20:38	118-79-6	
p-Terphenyl-d14 (S)	87	%	8-146		1	09/23/22 14:33	09/27/22 20:38	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	3.3	1		09/21/22 16:42	107-02-8	L1
Acrylonitrile	ND	ug/L	100	0.50	1		09/21/22 16:42	107-13-1	
Benzene	ND	ug/L	5.0	0.11	1		09/21/22 16:42	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.11	1		09/21/22 16:42	75-27-4	
Bromoform	ND	ug/L	5.0	0.036	1		09/21/22 16:42	75-25-2	
Bromomethane	ND	ug/L	5.0	0.19	1		09/21/22 16:42	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.065	1		09/21/22 16:42	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.098	1		09/21/22 16:42	108-90-7	
Chloroethane	ND	ug/L	5.0	0.17	1		09/21/22 16:42	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	0.37	1		09/21/22 16:42	110-75-8	
Chloroform	ND	ug/L	4.8	0.15	1		09/21/22 16:42	67-66-3	
Chloromethane	ND	ug/L	5.0	0.16	1		09/21/22 16:42	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.041	1		09/21/22 16:42	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.10	1		09/21/22 16:42	75-34-3	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

**Sample: 30-EFF-19SEP2022**      **Lab ID: 50326308007**      Collected: 09/19/22 16:20      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.12	1		09/21/22 16:42	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.13	1		09/21/22 16:42	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.13	1		09/21/22 16:42	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.12	1		09/21/22 16:42	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 16:42	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 16:42	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.082	1		09/21/22 16:42	100-41-4	
Methylene Chloride	ND	ug/L	5.0	1.6	1		09/21/22 16:42	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.090	1		09/21/22 16:42	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.16	1		09/21/22 16:42	127-18-4	
Toluene	ND	ug/L	5.0	0.11	1		09/21/22 16:42	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.097	1		09/21/22 16:42	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.11	1		09/21/22 16:42	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.12	1		09/21/22 16:42	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.11	1		09/21/22 16:42	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	95	%	91-114		1		09/21/22 16:42	1868-53-7	
4-Bromofluorobenzene (S)	97	%	85-120		1		09/21/22 16:42	460-00-4	
Toluene-d8 (S)	101	%	85-117		1		09/21/22 16:42	2037-26-5	

**335.4 Cyanide, Total**

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	<b>0.070</b>	mg/L	0.0050	0.0018	1	09/26/22 11:46	09/27/22 17:44	57-12-5	
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**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

**Sample: 30-DNS-19SEP2022**      **Lab ID: 50326308008**      Collected: 09/19/22 15:50      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**608.3 PCB**

Analytical Method: EPA 608.3 Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

PCB-1016 (Aroclor 1016)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:58	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:58	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:58	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:58	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:58	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.10	0.058	1	09/26/22 22:04	09/28/22 20:58	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.10	0.051	1	09/26/22 22:04	09/28/22 20:58	11096-82-5	

**Surrogates**

Tetrachloro-m-xylene (S)	66	%	1-123		1	09/26/22 22:04	09/28/22 20:58	877-09-8	
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**608.3 Pesticides**

Analytical Method: EPA 608.3 Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

Aldrin	ND	ug/L	0.050	0.0090	1	09/26/22 22:04	10/10/22 15:09	309-00-2	
alpha-BHC	ND	ug/L	0.050	0.011	1	09/26/22 22:04	10/10/22 15:09	319-84-6	
beta-BHC	ND	ug/L	0.050	0.014	1	09/26/22 22:04	10/10/22 15:09	319-85-7	
delta-BHC	ND	ug/L	0.050	0.013	1	09/26/22 22:04	10/10/22 15:09	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.050	0.012	1	09/26/22 22:04	10/10/22 15:09	58-89-9	
Chlordane (Technical)	ND	ug/L	0.50	0.27	1	09/26/22 22:04	10/10/22 15:09	57-74-9	
4,4'-DDD	ND	ug/L	0.10	0.024	1	09/26/22 22:04	10/10/22 15:09	72-54-8	
4,4'-DDE	ND	ug/L	0.10	0.018	1	09/26/22 22:04	10/10/22 15:09	72-55-9	
4,4'-DDT	ND	ug/L	0.10	0.036	1	09/26/22 22:04	10/10/22 15:09	50-29-3	
Dieldrin	ND	ug/L	0.10	0.022	1	09/26/22 22:04	10/10/22 15:09	60-57-1	
Endosulfan I	ND	ug/L	0.050	0.012	1	09/26/22 22:04	10/10/22 15:09	959-98-8	
Endosulfan II	ND	ug/L	0.10	0.025	1	09/26/22 22:04	10/10/22 15:09	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.10	0.021	1	09/26/22 22:04	10/10/22 15:09	1031-07-8	
Endrin	ND	ug/L	0.10	0.027	1	09/26/22 22:04	10/10/22 15:09	72-20-8	
Endrin aldehyde	ND	ug/L	0.10	0.025	1	09/26/22 22:04	10/10/22 15:09	7421-93-4	
Heptachlor	ND	ug/L	0.050	0.010	1	09/26/22 22:04	10/10/22 15:09	76-44-8	
Heptachlor epoxide	ND	ug/L	0.050	0.011	1	09/26/22 22:04	10/10/22 15:09	1024-57-3	
Toxaphene	ND	ug/L	1.0	0.36	1	09/26/22 22:04	10/10/22 15:09	8001-35-2	

**Surrogates**

Decachlorobiphenyl (S)	45	%	1-140		1	09/26/22 22:04	10/10/22 15:09	2051-24-3	
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**200.8 Metals, Total ICMS**

Analytical Method: EPA 200.8 Preparation Method: EPA 200.8  
Pace Analytical Services - Indianapolis

Antimony	<b>0.00088J</b>	mg/L	0.0010	0.00013	1	09/28/22 08:00	10/03/22 14:45	7440-36-0	
Arsenic	<b>0.0034</b>	mg/L	0.0010	0.00011	1	09/28/22 08:00	10/03/22 14:45	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	09/28/22 08:00	10/03/22 14:45	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	09/28/22 08:00	10/03/22 14:45	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	09/28/22 08:00	10/03/22 14:45	7440-47-3	
Copper	<b>0.0057</b>	mg/L	0.0010	0.00037	1	09/28/22 08:00	10/03/22 14:45	7440-50-8	
Lead	<b>0.00013J</b>	mg/L	0.0010	0.000080	1	09/28/22 08:00	10/03/22 14:45	7439-92-1	
Nickel	<b>0.0039</b>	mg/L	0.00050	0.00039	1	09/28/22 08:00	10/03/22 14:45	7440-02-0	
Selenium	<b>0.054</b>	mg/L	0.0010	0.00035	1	09/28/22 08:00	10/03/22 14:45	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	09/28/22 08:00	10/03/22 14:45	7440-22-4	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 30-DNS-19SEP2022 Lab ID: 50326308008 Collected: 09/19/22 15:50 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	0.000091J	mg/L	0.0010	0.000073	1	09/28/22 08:00	10/03/22 14:45	7440-28-0	
Zinc	0.0086	mg/L	0.0030	0.0010	1	09/28/22 08:00	10/03/22 14:45	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/03/22 09:26	10/03/22 13:28	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	10.0	1.8	1	09/23/22 14:33	09/27/22 20:54	83-32-9	
Acenaphthylene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:54	208-96-8	
Anthracene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:54	120-12-7	
Benzdine	ND	ug/L	50.0	6.0	1	09/23/22 14:33	09/27/22 20:54	92-87-5	
Benzo(a)anthracene	ND	ug/L	10.0	1.7	1	09/23/22 14:33	09/27/22 20:54	56-55-3	
Benzo(a)pyrene	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:54	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:54	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	10.0	2.2	1	09/23/22 14:33	09/27/22 20:54	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:54	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 20:54	101-55-3	
Butylbenzylphthalate	ND	ug/L	10.0	3.7	1	09/23/22 14:33	09/27/22 20:54	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	20.0	3.0	1	09/23/22 14:33	09/27/22 20:54	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	10.0	3.1	1	09/23/22 14:33	09/27/22 20:54	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	10.0	2.2	1	09/23/22 14:33	09/27/22 20:54	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	10.0	3.6	1	09/23/22 14:33	09/27/22 20:54	108-60-1	
2-Chloronaphthalene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:54	91-58-7	
2-Chlorophenol	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:54	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	10.0	2.7	1	09/23/22 14:33	09/27/22 20:54	7005-72-3	
Chrysene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:54	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:54	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	10.0	2.7	1	09/23/22 14:33	09/27/22 20:54	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 20:54	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 20:54	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	20.0	3.1	1	09/23/22 14:33	09/27/22 20:54	91-94-1	
2,4-Dichlorophenol	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:54	120-83-2	
Diethylphthalate	ND	ug/L	10.0	3.2	1	09/23/22 14:33	09/27/22 20:54	84-66-2	
2,4-Dimethylphenol	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:54	105-67-9	
Dimethylphthalate	ND	ug/L	10.0	2.3	1	09/23/22 14:33	09/27/22 20:54	131-11-3	
Di-n-butylphthalate	ND	ug/L	10.0	3.3	1	09/23/22 14:33	09/27/22 20:54	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	50.0	8.2	1	09/23/22 14:33	09/27/22 20:54	534-52-1	
2,4-Dinitrophenol	ND	ug/L	50.0	5.2	1	09/23/22 14:33	09/27/22 20:54	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	10.0	2.4	1	09/23/22 14:33	09/27/22 20:54	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	10.0	2.9	1	09/23/22 14:33	09/27/22 20:54	606-20-2	
Di-n-octylphthalate	ND	ug/L	10.0	7.3	1	09/23/22 14:33	09/27/22 20:54	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	10.0	2.1	1	09/23/22 14:33	09/27/22 20:54	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	5.0	4.2	1	09/23/22 14:33	09/27/22 20:54	117-81-7	

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### ANALYTICAL RESULTS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Sample: 30-DNS-19SEP2022 Lab ID: 50326308008 Collected: 09/19/22 15:50 Received: 09/20/22 12:15 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	10.0	1.8	1	09/23/22 14:33	09/27/22 20:54	206-44-0	
Fluorene	ND	ug/L	10.0	2.0	1	09/23/22 14:33	09/27/22 20:54	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	10.0	3.8	1	09/23/22 14:33	09/27/22 20:54	87-68-3	
Hexachlorobenzene	ND	ug/L	10.0	3.3	1	09/23/22 14:33	09/27/22 20:54	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	20.0	4.5	1	09/23/22 14:33	09/27/22 20:54	77-47-4	N2
Hexachloroethane	ND	ug/L	10.0	3.4	1	09/23/22 14:33	09/27/22 20:54	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	10.0	2.6	1	09/23/22 14:33	09/27/22 20:54	193-39-5	
Isophorone	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:54	78-59-1	
Naphthalene	ND	ug/L	10.0	2.4	1	09/23/22 14:33	09/27/22 20:54	91-20-3	
Nitrobenzene	ND	ug/L	10.0	3.1	1	09/23/22 14:33	09/27/22 20:54	98-95-3	
2-Nitrophenol	ND	ug/L	10.0	4.2	1	09/23/22 14:33	09/27/22 20:54	88-75-5	
4-Nitrophenol	ND	ug/L	50.0	5.1	1	09/23/22 14:33	09/27/22 20:54	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	20.0	3.5	1	09/23/22 14:33	09/27/22 20:54	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	10.0	3.0	1	09/23/22 14:33	09/27/22 20:54	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	10.0	2.2	1	09/23/22 14:33	09/27/22 20:54	86-30-6	
Pentachlorophenol	ND	ug/L	50.0	6.7	1	09/23/22 14:33	09/27/22 20:54	87-86-5	
Phenanthrene	ND	ug/L	10.0	1.9	1	09/23/22 14:33	09/27/22 20:54	85-01-8	
Phenol	ND	ug/L	10.0	1.2	1	09/23/22 14:33	09/27/22 20:54	108-95-2	
Pyrene	ND	ug/L	10.0	2.0	1	09/23/22 14:33	09/27/22 20:54	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	10.0	3.7	1	09/23/22 14:33	09/27/22 20:54	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	10.0	2.8	1	09/23/22 14:33	09/27/22 20:54	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	51	%	9-74		1	09/23/22 14:33	09/27/22 20:54	367-12-4	
Phenol-d5 (S)	31	%	8-424		1	09/23/22 14:33	09/27/22 20:54	4165-62-2	
Nitrobenzene-d5 (S)	82	%	15-314		1	09/23/22 14:33	09/27/22 20:54	4165-60-0	
2-Fluorobiphenyl (S)	73	%	32-92		1	09/23/22 14:33	09/27/22 20:54	321-60-8	
2,4,6-Tribromophenol (S)	89	%	27-125		1	09/23/22 14:33	09/27/22 20:54	118-79-6	
p-Terphenyl-d14 (S)	91	%	8-146		1	09/23/22 14:33	09/27/22 20:54	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	3.3	1		09/21/22 17:11	107-02-8	L1
Acrylonitrile	ND	ug/L	100	0.50	1		09/21/22 17:11	107-13-1	
Benzene	ND	ug/L	5.0	0.11	1		09/21/22 17:11	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.11	1		09/21/22 17:11	75-27-4	
Bromoform	ND	ug/L	5.0	0.036	1		09/21/22 17:11	75-25-2	
Bromomethane	ND	ug/L	5.0	0.19	1		09/21/22 17:11	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.065	1		09/21/22 17:11	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.098	1		09/21/22 17:11	108-90-7	
Chloroethane	ND	ug/L	5.0	0.17	1		09/21/22 17:11	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	0.37	1		09/21/22 17:11	110-75-8	
Chloroform	ND	ug/L	4.8	0.15	1		09/21/22 17:11	67-66-3	
Chloromethane	ND	ug/L	5.0	0.16	1		09/21/22 17:11	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.041	1		09/21/22 17:11	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.10	1		09/21/22 17:11	75-34-3	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

**Sample: 30-DNS-19SEP2022**      **Lab ID: 50326308008**      Collected: 09/19/22 15:50      Received: 09/20/22 12:15      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.12	1		09/21/22 17:11	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.13	1		09/21/22 17:11	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.13	1		09/21/22 17:11	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.12	1		09/21/22 17:11	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 17:11	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.057	1		09/21/22 17:11	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.082	1		09/21/22 17:11	100-41-4	
Methylene Chloride	ND	ug/L	5.0	1.6	1		09/21/22 17:11	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.090	1		09/21/22 17:11	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.16	1		09/21/22 17:11	127-18-4	
Toluene	ND	ug/L	5.0	0.11	1		09/21/22 17:11	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.097	1		09/21/22 17:11	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.11	1		09/21/22 17:11	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.12	1		09/21/22 17:11	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.11	1		09/21/22 17:11	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	95	%	91-114		1		09/21/22 17:11	1868-53-7	
4-Bromofluorobenzene (S)	97	%	85-120		1		09/21/22 17:11	460-00-4	
Toluene-d8 (S)	101	%	85-117		1		09/21/22 17:11	2037-26-5	

**335.4 Cyanide, Total**

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	ND	mg/L	0.0050	0.0018	1	09/26/22 11:46	09/27/22 17:45	57-12-5	
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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

QC Batch: 697565

Analysis Method: EPA 245.1

QC Batch Method: EPA 245.1

Analysis Description: 245.1 Mercury

Laboratory: Pace Analytical Services - Indianapolis

Associated Lab Samples: 50326308001

METHOD BLANK: 3207540

Matrix: Water

Associated Lab Samples: 50326308001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mercury	mg/L	ND	0.00020	0.00012	09/27/22 11:04	

LABORATORY CONTROL SAMPLE: 3207541

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mercury	mg/L	0.005	0.0051	102	85-115	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3207542 3207543

Parameter	Units	50325848001		3207542		3207543		% Rec Limits	RPD	Max RPD	Qual
		MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec				
Mercury	mg/L	ND	0.005	0.005	0.0049	0.0052	97	104	70-130	6	20

MATRIX SPIKE SAMPLE: 3207544

Parameter	Units	50326308001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Mercury	mg/L	ND	0.005	0.0052	103	70-130	

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**REPORT OF LABORATORY ANALYSIS**

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch: 698406	Analysis Method: EPA 245.1
QC Batch Method: EPA 245.1	Analysis Description: 245.1 Mercury
	Laboratory: Pace Analytical Services - Indianapolis

Associated Lab Samples: 50326308002, 50326308003, 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

METHOD BLANK: 3211279 Matrix: Water  
Associated Lab Samples: 50326308002, 50326308003, 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mercury	mg/L	ND	0.00020	0.00012	10/03/22 12:56	

LABORATORY CONTROL SAMPLE: 3211280

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mercury	mg/L	0.005	0.0051	102	85-115	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3211281 3211282

Parameter	Units	50326308007 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mercury	mg/L	ND	0.005	0.005	0.0049	0.0050	98	101	70-130	2	20	

MATRIX SPIKE SAMPLE: 3211283

Parameter	Units	50327098008 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Mercury	mg/L	ND	0.005	0.0050	100	70-130	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch: 697950 Analysis Method: EPA 200.8  
QC Batch Method: EPA 200.8 Analysis Description: 200.8 MET  
Laboratory: Pace Analytical Services - Indianapolis  
Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

METHOD BLANK: 3208912 Matrix: Water  
Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Antimony	mg/L	ND	0.0010	0.00013	10/03/22 13:56	
Arsenic	mg/L	ND	0.0010	0.00011	10/03/22 13:56	
Beryllium	mg/L	ND	0.00020	0.000033	10/03/22 13:56	
Cadmium	mg/L	ND	0.00020	0.000034	10/03/22 13:56	
Chromium	mg/L	ND	0.0020	0.00063	10/03/22 13:56	
Copper	mg/L	ND	0.0010	0.00037	10/03/22 13:56	
Lead	mg/L	ND	0.0010	0.000080	10/03/22 13:56	
Nickel	mg/L	ND	0.00050	0.00039	10/03/22 13:56	
Selenium	mg/L	ND	0.0010	0.00035	10/03/22 13:56	
Silver	mg/L	ND	0.00050	0.00037	10/03/22 13:56	
Thallium	mg/L	ND	0.0010	0.000073	10/03/22 13:56	
Zinc	mg/L	ND	0.0030	0.0010	10/03/22 13:56	

LABORATORY CONTROL SAMPLE: 3208913

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Antimony	mg/L	0.04	0.042	105	85-115	
Arsenic	mg/L	0.04	0.040	100	85-115	
Beryllium	mg/L	0.04	0.041	104	85-115	
Cadmium	mg/L	0.04	0.039	98	85-115	
Chromium	mg/L	0.04	0.041	103	85-115	
Copper	mg/L	0.04	0.040	99	85-115	
Lead	mg/L	0.04	0.041	102	85-115	
Nickel	mg/L	0.04	0.039	98	85-115	
Selenium	mg/L	0.04	0.040	101	85-115	
Silver	mg/L	0.04	0.041	103	85-115	
Thallium	mg/L	0.04	0.042	105	85-115	
Zinc	mg/L	0.04	0.040	100	85-115	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3208914 3208915

Parameter	Units	MS		MSD		MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
		Spike Conc.	Spike Conc.	Result	Result						
Antimony	mg/L	<0.0010	0.04	0.04	0.043	0.043	108	107	70-130	1	20
Arsenic	mg/L	<0.0010	0.04	0.04	0.040	0.040	99	99	70-130	1	20
Beryllium	mg/L	<0.00020	0.04	0.04	0.040	0.040	100	101	70-130	1	20

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3208914											
Parameter	Units	50326724002 Result	MS		MSD		MS % Rec	MSD % Rec	% Rec Limits	Max RPD	Qual
			Spike Conc.	MSD Conc.	MS Result	MSD Result					
Cadmium	mg/L	<0.00020	0.04	0.04	0.038	0.038	95	96	70-130	1	20
Chromium	mg/L	<0.0020	0.04	0.04	0.041	0.042	101	103	70-130	2	20
Copper	mg/L	0.026	0.04	0.04	0.062	0.063	88	90	70-130	2	20
Lead	mg/L	<0.0010	0.04	0.04	0.041	0.042	101	103	70-130	2	20
Nickel	mg/L	0.0010	0.04	0.04	0.037	0.037	90	91	70-130	1	20
Selenium	mg/L	<0.0010	0.04	0.04	0.042	0.041	103	102	70-130	1	20
Silver	mg/L	<0.00050	0.04	0.04	0.040	0.039	100	98	70-130	3	20
Thallium	mg/L	<0.0010	0.04	0.04	0.043	0.043	107	108	70-130	1	20
Zinc	mg/L	0.0054	0.04	0.04	0.042	0.042	91	92	70-130	1	20

MATRIX SPIKE SAMPLE: 3208916							
Parameter	Units	50326812001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Antimony	mg/L	0.00042J	0.04	0.045	112	70-130	
Arsenic	mg/L	0.0080	0.04	0.049	103	70-130	
Beryllium	mg/L	ND	0.04	0.041	103	70-130	
Cadmium	mg/L	ND	0.04	0.039	99	70-130	
Chromium	mg/L	0.00099J	0.04	0.040	98	70-130	
Copper	mg/L	0.0014	0.04	0.038	92	70-130	
Lead	mg/L	0.00040J	0.04	0.043	107	70-130	
Nickel	mg/L	0.0048	0.04	0.041	91	70-130	
Selenium	mg/L	0.053	0.04	0.096	107	70-130	
Silver	mg/L	ND	0.04	0.040	100	70-130	
Thallium	mg/L	ND	0.04	0.044	110	70-130	
Zinc	mg/L	0.037	0.04	0.074	92	70-130	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch: 696949 Analysis Method: EPA 624.1  
QC Batch Method: EPA 624.1 Analysis Description: 624.1 MSV  
Laboratory: Pace Analytical Services - Indianapolis  
Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

METHOD BLANK: 3204270 Matrix: Water  
Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
1,1,1-Trichloroethane	ug/L	ND	5.0	0.097	09/21/22 13:16	
1,1,2,2-Tetrachloroethane	ug/L	ND	5.0	0.090	09/21/22 13:16	
1,1,2-Trichloroethane	ug/L	ND	5.0	0.11	09/21/22 13:16	
1,1-Dichloroethane	ug/L	ND	5.0	0.10	09/21/22 13:16	
1,1-Dichloroethene	ug/L	ND	5.0	0.13	09/21/22 13:16	
1,2-Dichloroethane	ug/L	ND	5.0	0.12	09/21/22 13:16	
1,2-Dichloropropane	ug/L	ND	5.0	0.12	09/21/22 13:16	
2-Chloroethylvinyl ether	ug/L	ND	50.0	0.37	09/21/22 13:16	
Acrolein	ug/L	ND	50.0	3.3	09/21/22 13:16	
Acrylonitrile	ug/L	ND	100	0.50	09/21/22 13:16	
Benzene	ug/L	ND	5.0	0.11	09/21/22 13:16	
Bromodichloromethane	ug/L	ND	5.0	0.11	09/21/22 13:16	
Bromoform	ug/L	ND	5.0	0.036	09/21/22 13:16	
Bromomethane	ug/L	ND	5.0	0.19	09/21/22 13:16	
Carbon tetrachloride	ug/L	ND	5.0	0.065	09/21/22 13:16	
Chlorobenzene	ug/L	ND	5.0	0.098	09/21/22 13:16	
Chloroethane	ug/L	ND	5.0	0.17	09/21/22 13:16	
Chloroform	ug/L	ND	4.8	0.15	09/21/22 13:16	
Chloromethane	ug/L	ND	5.0	0.16	09/21/22 13:16	
cis-1,3-Dichloropropene	ug/L	ND	5.0	0.057	09/21/22 13:16	
Dibromochloromethane	ug/L	ND	5.0	0.041	09/21/22 13:16	
Ethylbenzene	ug/L	ND	5.0	0.082	09/21/22 13:16	
Methylene Chloride	ug/L	2.0J	5.0	1.6	09/21/22 13:16	
Tetrachloroethene	ug/L	ND	5.0	0.16	09/21/22 13:16	
Toluene	ug/L	ND	5.0	0.11	09/21/22 13:16	
trans-1,2-Dichloroethene	ug/L	ND	4.8	0.13	09/21/22 13:16	
trans-1,3-Dichloropropene	ug/L	ND	5.0	0.057	09/21/22 13:16	
Trichloroethene	ug/L	ND	5.0	0.12	09/21/22 13:16	
Vinyl chloride	ug/L	ND	2.0	0.11	09/21/22 13:16	
4-Bromofluorobenzene (S)	%	99	85-120		09/21/22 13:16	
Dibromofluoromethane (S)	%	96	91-114		09/21/22 13:16	
Toluene-d8 (S)	%	102	85-117		09/21/22 13:16	

LABORATORY CONTROL SAMPLE: 3204271

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
1,1,1-Trichloroethane	ug/L	20	20.6	103	70-130	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

LABORATORY CONTROL SAMPLE: 3204271

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
1,1,2,2-Tetrachloroethane	ug/L	20	23.7	119	60-140	
1,1,2-Trichloroethane	ug/L	20	22.2	111	70-130	
1,1-Dichloroethane	ug/L	20	22.4	112	70-130	
1,1-Dichloroethene	ug/L	20	24.4	122	50-150	
1,2-Dichloroethane	ug/L	20	20.9	104	70-130	
1,2-Dichloropropane	ug/L	20	22.3	111	35-165	
2-Chloroethylvinyl ether	ug/L	100	120	120	1-225	
Acrolein	ug/L	400	759	190	60-140 L1	
Acrylonitrile	ug/L	100	120	120	60-140	
Benzene	ug/L	20	21.3	106	65-135	
Bromodichloromethane	ug/L	20	20.8	104	65-135	
Bromoform	ug/L	20	19.0	95	70-130	
Bromomethane	ug/L	20	20.8	104	15-185	
Carbon tetrachloride	ug/L	20	19.6	98	70-130	
Chlorobenzene	ug/L	20	20.8	104	65-135	
Chloroethane	ug/L	20	28.2	141	40-160	
Chloroform	ug/L	20	20.7	103	70-135	
Chloromethane	ug/L	20	28.4	142	1-205	
cis-1,3-Dichloropropene	ug/L	20	21.5	107	25-175	
Dibromochloromethane	ug/L	20	19.8	99	70-135	
Ethylbenzene	ug/L	20	20.9	105	60-140	
Methylene Chloride	ug/L	20	19.2	96	60-140	
Tetrachloroethene	ug/L	20	20.0	100	70-130	
Toluene	ug/L	20	20.8	104	70-130	
trans-1,2-Dichloroethene	ug/L	20	21.0	105	70-130	
trans-1,3-Dichloropropene	ug/L	20	20.5	102	50-150	
Trichloroethene	ug/L	20	20.9	105	65-135	
Vinyl chloride	ug/L	20	28.3	142	5-195	
4-Bromofluorobenzene (S)	%			101	85-120	
Dibromofluoromethane (S)	%			98	91-114	
Toluene-d8 (S)	%			101	85-117	

MATRIX SPIKE SAMPLE: 3204272

Parameter	Units	50326348001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
1,1,1-Trichloroethane	ug/L	ND	20	23.5	117	52-162	
1,1,2,2-Tetrachloroethane	ug/L	ND	20	26.9	134	46-157	
1,1,2-Trichloroethane	ug/L	ND	20	25.5	127	52-150	
1,1-Dichloroethane	ug/L	ND	20	25.9	130	59-155	
1,1-Dichloroethene	ug/L	ND	20	28.6	143	1-234	
1,2-Dichloroethane	ug/L	ND	20	23.7	118	49-155	
1,2-Dichloropropane	ug/L	ND	20	25.6	128	1-210	
2-Chloroethylvinyl ether	ug/L	ND	100	ND	0	1-305 M1	
Acrolein	ug/L	ND	400	833	208	40-160 M0	
Acrylonitrile	ug/L	ND	100	138	138	40-160	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

MATRIX SPIKE SAMPLE: 3204272		50326348001	Spike	MS	MS	% Rec	
Parameter	Units	Result	Conc.	Result	% Rec	Limits	Qualifiers
Benzene	ug/L	ND	20	24.7	124	37-151	
Bromodichloromethane	ug/L	ND	20	23.3	116	35-155	
Bromoform	ug/L	ND	20	21.2	106	45-169	
Bromomethane	ug/L	ND	20	24.2	121	1-242	
Carbon tetrachloride	ug/L	ND	20	22.3	111	70-140	
Chlorobenzene	ug/L	ND	20	23.8	119	37-160	
Chloroethane	ug/L	ND	20	33.6	168	14-230	
Chloroform	ug/L	ND	20	23.6	118	51-138	
Chloromethane	ug/L	ND	20	33.6	168	1-273	
cis-1,3-Dichloropropene	ug/L	ND	20	23.6	118	1-227	
Dibromochloromethane	ug/L	ND	20	22.4	112	53-149	
Ethylbenzene	ug/L	ND	20	23.2	116	37-162	
Methylene Chloride	ug/L	ND	20	20.0	100	1-221	
Tetrachloroethene	ug/L	ND	20	21.3	106	64-148	
Toluene	ug/L	ND	20	23.8	119	47-150	
trans-1,2-Dichloroethene	ug/L	ND	20	23.6	118	54-156	
trans-1,3-Dichloropropene	ug/L	ND	20	22.5	113	17-183	
Trichloroethene	ug/L	ND	20	23.6	118	70-157	
Vinyl chloride	ug/L	ND	20	33.3	166	1-251	
4-Bromofluorobenzene (S)	%				101	85-120	
Dibromofluoromethane (S)	%				96	91-114	
Toluene-d8 (S)	%				103	85-117	

SAMPLE DUPLICATE: 3204273

Parameter	Units	50326347001	Dup	RPD	Max	Qualifiers
		Result	Result		RPD	
1,1,1-Trichloroethane	ug/L	ND	ND		36	
1,1,2,2-Tetrachloroethane	ug/L	ND	ND		61	
1,1,2-Trichloroethane	ug/L	ND	ND		45	
1,1-Dichloroethane	ug/L	ND	ND		40	
1,1-Dichloroethene	ug/L	ND	ND		32	
1,2-Dichloroethane	ug/L	ND	ND		49	
1,2-Dichloropropane	ug/L	ND	ND		55	
2-Chloroethylvinyl ether	ug/L	ND	ND		71	
Acrolein	ug/L	ND	ND		60	
Acrylonitrile	ug/L	ND	ND		60	
Benzene	ug/L	ND	ND		61	
Bromodichloromethane	ug/L	ND	ND		56	
Bromoform	ug/L	ND	ND		42	
Bromomethane	ug/L	ND	ND		61	
Carbon tetrachloride	ug/L	ND	ND		41	
Chlorobenzene	ug/L	ND	ND		53	
Chloroethane	ug/L	ND	ND		78	
Chloroform	ug/L	ND	ND		54	
Chloromethane	ug/L	ND	ND		60	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

SAMPLE DUPLICATE: 3204273

Parameter	Units	50326347001 Result	Dup Result	RPD	Max RPD	Qualifiers
cis-1,3-Dichloropropene	ug/L	ND	ND		58	
Dibromochloromethane	ug/L	ND	ND		50	
Ethylbenzene	ug/L	ND	ND		63	
Methylene Chloride	ug/L	ND	ND		28	
Tetrachloroethene	ug/L	ND	ND		39	
Toluene	ug/L	ND	ND		41	
trans-1,2-Dichloroethene	ug/L	ND	ND		45	
trans-1,3-Dichloropropene	ug/L	ND	ND		86	
Trichloroethene	ug/L	ND	ND		48	
Vinyl chloride	ug/L	ND	ND		66	
4-Bromofluorobenzene (S)	%.	98	99			
Dibromofluoromethane (S)	%.	95	95			
Toluene-d8 (S)	%.	101	101			

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch: 697274 Analysis Method: EPA 608.3  
QC Batch Method: EPA 608.3 Analysis Description: 608.3 PCB  
Laboratory: Pace Analytical Services - Indianapolis  
Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006

METHOD BLANK: 3205875 Matrix: Water  
Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
PCB-1016 (Aroclor 1016)	ug/L	ND	0.10	0.058	09/26/22 20:01	
PCB-1221 (Aroclor 1221)	ug/L	ND	0.10	0.058	09/26/22 20:01	
PCB-1232 (Aroclor 1232)	ug/L	ND	0.10	0.058	09/26/22 20:01	
PCB-1242 (Aroclor 1242)	ug/L	ND	0.10	0.058	09/26/22 20:01	
PCB-1248 (Aroclor 1248)	ug/L	ND	0.10	0.058	09/26/22 20:01	
PCB-1254 (Aroclor 1254)	ug/L	ND	0.10	0.058	09/26/22 20:01	
PCB-1260 (Aroclor 1260)	ug/L	ND	0.10	0.051	09/26/22 20:01	
Tetrachloro-m-xylene (S)	%	37	1-123		09/26/22 20:01	

LABORATORY CONTROL SAMPLE: 3205876

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
PCB-1016 (Aroclor 1016)	ug/L	0.5	0.46	92	50-140	
PCB-1260 (Aroclor 1260)	ug/L	0.5	0.40	79	8-140	
Tetrachloro-m-xylene (S)	%			27	1-123	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3205877 3205878

Parameter	Units	50326349001		3205877		3205878		% Rec	% Rec	Limits	RPD	Max RPD	Qual
		MS Result	MSD Spike Conc.	MS Spike Conc.	MSD Result	MS Result	MSD Result						
PCB-1016 (Aroclor 1016)	ug/L	ND	1.1	1.1	0.74	0.83	70	79	50-140	12	36		
PCB-1260 (Aroclor 1260)	ug/L	ND	1.1	1.1	0.65	0.70	62	66	8-140	7	38		
Tetrachloro-m-xylene (S)	%						33	33	1-123				

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch: 697778	Analysis Method: EPA 608.3
QC Batch Method: EPA 608.3	Analysis Description: 608.3 PCB
	Laboratory: Pace Analytical Services - Indianapolis

Associated Lab Samples: 50326308007, 50326308008

METHOD BLANK: 3208232 Matrix: Water

Associated Lab Samples: 50326308007, 50326308008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
PCB-1016 (Aroclor 1016)	ug/L	ND	0.10	0.058	09/28/22 20:14	
PCB-1221 (Aroclor 1221)	ug/L	ND	0.10	0.058	09/28/22 20:14	
PCB-1232 (Aroclor 1232)	ug/L	ND	0.10	0.058	09/28/22 20:14	
PCB-1242 (Aroclor 1242)	ug/L	ND	0.10	0.058	09/28/22 20:14	
PCB-1248 (Aroclor 1248)	ug/L	ND	0.10	0.058	09/28/22 20:14	
PCB-1254 (Aroclor 1254)	ug/L	ND	0.10	0.058	09/28/22 20:14	
PCB-1260 (Aroclor 1260)	ug/L	ND	0.10	0.051	09/28/22 20:14	
Tetrachloro-m-xylene (S)	%	55	1-123		09/28/22 20:14	

LABORATORY CONTROL SAMPLE: 3208233

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
PCB-1016 (Aroclor 1016)	ug/L	0.5	0.47	93	50-140	
PCB-1260 (Aroclor 1260)	ug/L	0.5	0.39	77	8-140	
Tetrachloro-m-xylene (S)	%			68	1-123	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3208240 3208241

Parameter	Units	MS		MSD		MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
		50326210003 Result	Spike Conc.	Spike Conc.	Result						
PCB-1016 (Aroclor 1016)	ug/L	ND	1	1	0.58	0.53	58	53	50-140	10	36
PCB-1260 (Aroclor 1260)	ug/L	ND	1	1	0.49	0.40	49	40	8-140	19	38
Tetrachloro-m-xylene (S)	%						40	36	1-123		

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch: 697276 Analysis Method: EPA 608.3  
QC Batch Method: EPA 608.3 Analysis Description: 608.3 Pesticides  
Laboratory: Pace Analytical Services - Indianapolis  
Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006

METHOD BLANK: 3205889 Matrix: Water  
Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
4,4'-DDD	ug/L	ND	0.10	0.024	10/04/22 15:08	
4,4'-DDE	ug/L	ND	0.10	0.018	10/04/22 15:08	
4,4'-DDT	ug/L	ND	0.10	0.036	10/04/22 15:08	
Aldrin	ug/L	ND	0.050	0.0090	10/04/22 15:08	
alpha-BHC	ug/L	ND	0.050	0.011	10/04/22 15:08	
beta-BHC	ug/L	ND	0.050	0.014	10/04/22 15:08	
Chlordane (Technical)	ug/L	ND	0.50	0.27	10/04/22 15:08	
delta-BHC	ug/L	ND	0.050	0.013	10/04/22 15:08	
Dieldrin	ug/L	ND	0.10	0.022	10/04/22 15:08	
Endosulfan I	ug/L	ND	0.050	0.012	10/04/22 15:08	
Endosulfan II	ug/L	ND	0.10	0.025	10/04/22 15:08	
Endosulfan sulfate	ug/L	ND	0.10	0.021	10/04/22 15:08	
Endrin	ug/L	ND	0.10	0.027	10/04/22 15:08	
Endrin aldehyde	ug/L	ND	0.10	0.025	10/04/22 15:20	
gamma-BHC (Lindane)	ug/L	ND	0.050	0.012	10/04/22 15:08	
Heptachlor	ug/L	ND	0.050	0.010	10/04/22 15:08	
Heptachlor epoxide	ug/L	ND	0.050	0.011	10/04/22 15:08	
Toxaphene	ug/L	ND	1.0	0.36	10/04/22 15:08	
Decachlorobiphenyl (S)	%	78	1-140		10/04/22 15:08	

LABORATORY CONTROL SAMPLE: 3205890

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
4,4'-DDD	ug/L	0.2	0.15	75	31-141	
4,4'-DDE	ug/L	0.2	0.11	57	30-145	
4,4'-DDT	ug/L	0.2	0.13	66	25-160	
Aldrin	ug/L	0.1	0.018J	18	42-140	L2
alpha-BHC	ug/L	0.1	0.071	71	37-140	
beta-BHC	ug/L	0.1	0.091	91	17-147	
delta-BHC	ug/L	0.1	0.046J	46	19-140	
Dieldrin	ug/L	0.2	0.15	74	36-146	
Endosulfan I	ug/L	0.1	0.074	74	45-153	
Endosulfan II	ug/L	0.2	0.15	73	1-202	
Endosulfan sulfate	ug/L	0.2	0.13	63	26-144	
Endrin	ug/L	0.2	0.15	77	30-147	
Endrin aldehyde	ug/L	0.2	0.17	86	42-161	
gamma-BHC (Lindane)	ug/L	0.1	0.074	74	32-140	
Heptachlor	ug/L	0.1	0.033J	33	34-140	L2
Heptachlor epoxide	ug/L	0.1	0.077	77	37-142	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

LABORATORY CONTROL SAMPLE: 3205890

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Decachlorobiphenyl (S)	%.			53	1-140	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3205891 3205892

Parameter	Units	MS		MSD		MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
		50326349002 Result	Spike Conc.	Spike Conc.	Result						
4,4'-DDD	ug/L	ND	0.45	0.45	0.32	0.28	71	62	31-141	13	39
4,4'-DDE	ug/L	ND	0.45	0.45	0.35	0.31	77	69	30-145	11	35
4,4'-DDT	ug/L	ND	0.45	0.45	0.22J	0.19J	49	43	25-160		42
Aldrin	ug/L	ND	0.22	0.22	0.065J	0.065J	29	29	42-140		35 M0
alpha-BHC	ug/L	ND	0.22	0.22	0.19	0.18	86	82	37-140	5	36
beta-BHC	ug/L	ND	0.22	0.22	0.23	0.22	100	98	17-147	2	44
delta-BHC	ug/L	ND	0.22	0.22	0.075J	0.071J	33	32	19-140		52
Dieldrin	ug/L	ND	0.45	0.45	0.35	0.33	77	74	36-146	4	49
Endosulfan I	ug/L	ND	0.22	0.22	0.18	0.17	80	76	45-153	5	28
Endosulfan II	ug/L	ND	0.45	0.45	0.28	0.27	63	61	1-202	3	53
Endosulfan sulfate	ug/L	ND	0.45	0.45	0.19J	0.17J	42	38	26-144		38
Endrin	ug/L	ND	0.45	0.45	0.35	0.34	78	75	30-147	3	48
Endrin aldehyde	ug/L	ND	0.45	0.45	ND	ND	120	115	1-179		30
gamma-BHC (Lindane)	ug/L	ND	0.22	0.22	0.19	0.18	83	80	32-140	3	35
Heptachlor	ug/L	ND	0.22	0.22	0.11	0.11J	51	48	34-140		43
Heptachlor epoxide	ug/L	ND	0.22	0.22	0.19	0.19	85	84	37-142	1	26
Decachlorobiphenyl (S)	%.						37	48	1-140		

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch: 697779 Analysis Method: EPA 608.3  
QC Batch Method: EPA 608.3 Analysis Description: 608.3 Pesticides  
Laboratory: Pace Analytical Services - Indianapolis

Associated Lab Samples: 50326308007, 50326308008

METHOD BLANK: 3208236 Matrix: Water

Associated Lab Samples: 50326308007, 50326308008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
4,4'-DDD	ug/L	ND	0.10	0.024	10/05/22 21:55	
4,4'-DDE	ug/L	ND	0.10	0.018	10/05/22 21:55	
4,4'-DDT	ug/L	ND	0.10	0.036	10/05/22 21:55	
Aldrin	ug/L	ND	0.050	0.0090	10/05/22 21:55	
alpha-BHC	ug/L	ND	0.050	0.011	10/05/22 21:55	
beta-BHC	ug/L	ND	0.050	0.014	10/05/22 21:55	
Chlordane (Technical)	ug/L	ND	0.50	0.27	10/05/22 21:55	
delta-BHC	ug/L	ND	0.050	0.013	10/05/22 21:55	
Dieldrin	ug/L	ND	0.10	0.022	10/05/22 21:55	
Endosulfan I	ug/L	ND	0.050	0.012	10/05/22 21:55	
Endosulfan II	ug/L	ND	0.10	0.025	10/05/22 21:55	
Endosulfan sulfate	ug/L	ND	0.10	0.021	10/05/22 21:55	
Endrin	ug/L	ND	0.10	0.027	10/05/22 21:55	
Endrin aldehyde	ug/L	ND	0.10	0.025	10/05/22 21:55	
gamma-BHC (Lindane)	ug/L	ND	0.050	0.012	10/05/22 21:55	
Heptachlor	ug/L	ND	0.050	0.010	10/05/22 21:55	
Heptachlor epoxide	ug/L	ND	0.050	0.011	10/05/22 21:55	
Toxaphene	ug/L	ND	1.0	0.36	10/05/22 21:55	
Decachlorobiphenyl (S)	%	49	1-140		10/05/22 21:55	

LABORATORY CONTROL SAMPLE: 3208237

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
4,4'-DDD	ug/L	0.2	0.19	96	31-141	
4,4'-DDE	ug/L	0.2	0.18	89	30-145	
4,4'-DDT	ug/L	0.2	0.18	88	25-160	
Aldrin	ug/L	0.1	0.072	72	42-140	
alpha-BHC	ug/L	0.1	0.086	86	37-140	
beta-BHC	ug/L	0.1	0.092	92	17-147	
delta-BHC	ug/L	0.1	0.053	53	19-140	
Dieldrin	ug/L	0.2	0.20	102	36-146	
Endosulfan I	ug/L	0.1	0.092	92	45-153	
Endosulfan II	ug/L	0.2	0.19	93	1-202	
Endosulfan sulfate	ug/L	0.2	0.17	84	26-144	
Endrin	ug/L	0.2	0.19	94	30-147	
Endrin aldehyde	ug/L	0.2	0.21	107	42-161	
gamma-BHC (Lindane)	ug/L	0.1	0.088	88	32-140	
Heptachlor	ug/L	0.1	0.075	75	34-140	
Heptachlor epoxide	ug/L	0.1	0.097	97	37-142	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

LABORATORY CONTROL SAMPLE: 3208237

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Decachlorobiphenyl (S)	%.			58	1-140	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3208242 3208243

Parameter	Units	50326541001		MSD		MSD		% Rec	% Rec	% Rec	Limits	RPD	Max RPD	Qual
		Result	Spike Conc.	Spike Conc.	MS Result	MSD Result	MS % Rec							
4,4'-DDD	ug/L	<0.10	0.4	0.4	0.18J	0.19J	45	49	31-141		39			
4,4'-DDE	ug/L	<0.10	0.4	0.4	0.12J	0.12J	31	31	30-145		35			
4,4'-DDT	ug/L	<0.10	0.4	0.4	0.13J	0.11J	32	28	25-160		42			
Aldrin	ug/L	<0.050	0.2	0.2	0.064J	0.072J	32	36	42-140		35 M1			
alpha-BHC	ug/L	<0.050	0.2	0.2	0.070J	0.078J	35	39	37-140		36 M1			
beta-BHC	ug/L	<0.050	0.2	0.2	0.045J	0.045J	22	22	17-147		44			
delta-BHC	ug/L	<0.050	0.2	0.2	0.059J	0.062J	30	31	19-140		52			
Dieldrin	ug/L	<0.10	0.4	0.4	0.084J	0.11J	21	26	36-146		49 M1			
Endosulfan I	ug/L	<0.050	0.2	0.2	0.058J	0.058J	29	29	45-153		28 M1			
Endosulfan II	ug/L	<0.10	0.4	0.4	0.10J	0.11J	25	28	1-202		53			
Endosulfan sulfate	ug/L	<0.10	0.4	0.4	0.082J	0.11J	20	27	26-144		38 M1			
Endrin	ug/L	<0.10	0.4	0.4	0.11J	0.12J	28	31	30-147		48 M1			
Endrin aldehyde	ug/L	<0.10	0.4	0.4	0.055J	0.060J	14	15	1-179		30			
gamma-BHC (Lindane)	ug/L	<0.050	0.2	0.2	0.074J	0.072J	37	36	32-140		35			
Heptachlor	ug/L	<0.050	0.2	0.2	0.099J	0.047J	50	24	34-140		43 M1			
Heptachlor epoxide	ug/L	<0.050	0.2	0.2	0.055J	0.081J	27	40	37-142		26 M1			
Decachlorobiphenyl (S)	%.						12	27	1-140					

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**REPORT OF LABORATORY ANALYSIS**

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch: 697239 Analysis Method: EPA 625.1  
QC Batch Method: EPA 625.1 Analysis Description: 625.1 MSS  
Laboratory: Pace Analytical Services - Indianapolis

Associated Lab Samples: 50326308001, 50326308002, 50326308003

METHOD BLANK: 3205604 Matrix: Water

Associated Lab Samples: 50326308001, 50326308002, 50326308003

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
1,2,4-Trichlorobenzene	ug/L	ND	10.0	3.7	09/23/22 15:50	
1,2-Dichlorobenzene	ug/L	ND	10.0	2.7	09/23/22 15:50	
1,2-Diphenylhydrazine	ug/L	ND	10.0	2.1	09/23/22 15:50	N2
1,3-Dichlorobenzene	ug/L	ND	10.0	2.9	09/23/22 15:50	
1,4-Dichlorobenzene	ug/L	ND	10.0	2.9	09/23/22 15:50	
2,4,6-Trichlorophenol	ug/L	ND	10.0	2.8	09/23/22 15:50	
2,4-Dichlorophenol	ug/L	ND	10.0	2.3	09/23/22 15:50	
2,4-Dimethylphenol	ug/L	ND	10.0	2.3	09/23/22 15:50	
2,4-Dinitrophenol	ug/L	ND	50.0	5.2	09/23/22 15:50	
2,4-Dinitrotoluene	ug/L	ND	10.0	2.4	09/23/22 15:50	
2,6-Dinitrotoluene	ug/L	ND	10.0	2.9	09/23/22 15:50	
2-Chloronaphthalene	ug/L	ND	10.0	2.6	09/23/22 15:50	
2-Chlorophenol	ug/L	ND	10.0	2.3	09/23/22 15:50	
2-Nitrophenol	ug/L	ND	10.0	4.2	09/23/22 15:50	
3,3'-Dichlorobenzidine	ug/L	ND	20.0	3.1	09/23/22 15:50	
4,6-Dinitro-2-methylphenol	ug/L	ND	50.0	8.2	09/23/22 15:50	
4-Bromophenylphenyl ether	ug/L	ND	10.0	2.9	09/23/22 15:50	
4-Chloro-3-methylphenol	ug/L	ND	20.0	3.0	09/23/22 15:50	
4-Chlorophenylphenyl ether	ug/L	ND	10.0	2.7	09/23/22 15:50	
4-Nitrophenol	ug/L	ND	50.0	5.1	09/23/22 15:50	
Acenaphthene	ug/L	ND	10.0	1.8	09/23/22 15:50	
Acenaphthylene	ug/L	ND	10.0	1.9	09/23/22 15:50	
Anthracene	ug/L	ND	10.0	1.9	09/23/22 15:50	
Benzidine	ug/L	ND	50.0	6.0	09/23/22 15:50	
Benzo(a)anthracene	ug/L	ND	10.0	1.7	09/23/22 15:50	
Benzo(a)pyrene	ug/L	ND	10.0	2.3	09/23/22 15:50	
Benzo(b)fluoranthene	ug/L	ND	10.0	2.6	09/23/22 15:50	
Benzo(g,h,i)perylene	ug/L	ND	10.0	2.2	09/23/22 15:50	
Benzo(k)fluoranthene	ug/L	ND	10.0	2.6	09/23/22 15:50	
bis(2-Chloroethoxy)methane	ug/L	ND	10.0	3.1	09/23/22 15:50	
bis(2-Chloroethyl) ether	ug/L	ND	10.0	2.2	09/23/22 15:50	
bis(2-Chloroisopropyl) ether	ug/L	ND	10.0	3.6	09/23/22 15:50	
bis(2-Ethylhexyl)phthalate	ug/L	ND	5.0	4.2	09/23/22 15:50	
Butylbenzylphthalate	ug/L	ND	10.0	3.7	09/23/22 15:50	
Chrysene	ug/L	ND	10.0	1.9	09/23/22 15:50	
Di-n-butylphthalate	ug/L	ND	10.0	3.3	09/23/22 15:50	
Di-n-octylphthalate	ug/L	ND	10.0	7.3	09/23/22 15:50	
Dibenz(a,h)anthracene	ug/L	ND	10.0	2.6	09/23/22 15:50	
Diethylphthalate	ug/L	ND	10.0	3.2	09/23/22 15:50	
Dimethylphthalate	ug/L	ND	10.0	2.3	09/23/22 15:50	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

METHOD BLANK: 3205604 Matrix: Water  
Associated Lab Samples: 50326308001, 50326308002, 50326308003

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Fluoranthene	ug/L	ND	10.0	1.8	09/23/22 15:50	
Fluorene	ug/L	ND	10.0	2.0	09/23/22 15:50	
Hexachloro-1,3-butadiene	ug/L	ND	10.0	3.8	09/23/22 15:50	
Hexachlorobenzene	ug/L	ND	10.0	3.3	09/23/22 15:50	
Hexachlorocyclopentadiene	ug/L	ND	20.0	4.5	09/23/22 15:50	N2
Hexachloroethane	ug/L	ND	10.0	3.4	09/23/22 15:50	N2
Indeno(1,2,3-cd)pyrene	ug/L	ND	10.0	2.6	09/23/22 15:50	
Isophorone	ug/L	ND	10.0	1.9	09/23/22 15:50	
N-Nitroso-di-n-propylamine	ug/L	ND	10.0	3.0	09/23/22 15:50	
N-Nitrosodimethylamine	ug/L	ND	20.0	3.5	09/23/22 15:50	
N-Nitrosodiphenylamine	ug/L	ND	10.0	2.2	09/23/22 15:50	
Naphthalene	ug/L	ND	10.0	2.4	09/23/22 15:50	
Nitrobenzene	ug/L	ND	10.0	3.1	09/23/22 15:50	
Pentachlorophenol	ug/L	ND	50.0	6.7	09/23/22 15:50	
Phenanthrene	ug/L	ND	10.0	1.9	09/23/22 15:50	
Phenol	ug/L	ND	10.0	1.2	09/23/22 15:50	
Pyrene	ug/L	ND	10.0	2.0	09/23/22 15:50	
2,4,6-Tribromophenol (S)	%	81	27-125		09/23/22 15:50	
2-Fluorobiphenyl (S)	%	49	32-92		09/23/22 15:50	
2-Fluorophenol (S)	%	46	9-74		09/23/22 15:50	
Nitrobenzene-d5 (S)	%	69	15-314		09/23/22 15:50	
p-Terphenyl-d14 (S)	%	88	8-146		09/23/22 15:50	
Phenol-d5 (S)	%	36	8-424		09/23/22 15:50	

LABORATORY CONTROL SAMPLE: 3205605

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
1,2,4-Trichlorobenzene	ug/L	50	31.2	62	44-142	
1,2-Dichlorobenzene	ug/L	50	32.8	66	31-79	
1,2-Diphenylhydrazine	ug/L	50	51.0	102	59-111	N2
1,3-Dichlorobenzene	ug/L	50	28.5	57	28-73	
1,4-Dichlorobenzene	ug/L	50	28.8	58	29-76	
2,4,6-Trichlorophenol	ug/L	50	49.3	99	37-144	
2,4-Dichlorophenol	ug/L	50	48.6	97	39-135	
2,4-Dimethylphenol	ug/L	50	50.6	101	32-120	
2,4-Dinitrophenol	ug/L	50	29.6J	59	1-191	
2,4-Dinitrotoluene	ug/L	50	49.3	99	39-139	
2,6-Dinitrotoluene	ug/L	50	47.8	96	50-158	
2-Chloronaphthalene	ug/L	50	42.9	86	60-120	
2-Chlorophenol	ug/L	50	47.1	94	23-134	
2-Nitrophenol	ug/L	50	47.4	95	29-182	
3,3'-Dichlorobenzidine	ug/L	100	60.5	61	1-262	
4,6-Dinitro-2-methylphenol	ug/L	50	37.8J	76	1-181	
4-Bromophenylphenyl ether	ug/L	50	49.3	99	53-127	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

LABORATORY CONTROL SAMPLE: 3205605

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
4-Chloro-3-methylphenol	ug/L	50	49.7	99	22-147	
4-Chlorophenylphenyl ether	ug/L	50	49.9	100	25-158	
4-Nitrophenol	ug/L	50	24.5J	49	1-132	
Acenaphthene	ug/L	50	46.2	92	47-145	
Acenaphthylene	ug/L	50	43.8	88	33-145	
Anthracene	ug/L	50	50.4	101	27-133	
Benzidine	ug/L	100	ND	3	1-64	
Benzo(a)anthracene	ug/L	50	52.0	104	33-143	
Benzo(a)pyrene	ug/L	50	45.9	92	17-163	
Benzo(b)fluoranthene	ug/L	50	52.7	105	24-159	
Benzo(g,h,i)perylene	ug/L	50	47.4	95	1-219	
Benzo(k)fluoranthene	ug/L	50	47.1	94	11-162	
bis(2-Chloroethoxy)methane	ug/L	50	48.1	96	33-184	
bis(2-Chloroethyl) ether	ug/L	50	50.6	101	12-158	
bis(2-Chloroisopropyl) ether	ug/L	50	52.0	104	36-166	
bis(2-Ethylhexyl)phthalate	ug/L	50	59.8	120	8-158	
Butylbenzylphthalate	ug/L	50	60.5	121	1-152	
Chrysene	ug/L	50	53.5	107	17-168	
Di-n-butylphthalate	ug/L	50	52.3	105	1-120	
Di-n-octylphthalate	ug/L	50	58.4	117	4-146	
Dibenz(a,h)anthracene	ug/L	50	49.1	98	1-227	
Diethylphthalate	ug/L	50	49.6	99	1-120	
Dimethylphthalate	ug/L	50	49.0	98	1-120	
Fluoranthene	ug/L	50	47.9	96	26-137	
Fluorene	ug/L	50	48.9	98	59-121	
Hexachloro-1,3-butadiene	ug/L	50	26.4	53	24-120	
Hexachlorobenzene	ug/L	50	45.7	91	1-152	
Hexachlorocyclopentadiene	ug/L	50	26.2	52	5-92 N2	
Hexachloroethane	ug/L	50	27.5	55	40-120 N2	
Indeno(1,2,3-cd)pyrene	ug/L	50	50.1	100	1-171	
Isophorone	ug/L	50	45.8	92	21-196	
N-Nitroso-di-n-propylamine	ug/L	50	50.2	100	1-230	
N-Nitrosodimethylamine	ug/L	50	30.1	60	1-107	
N-Nitrosodiphenylamine	ug/L	50	50.3	101	65-108	
Naphthalene	ug/L	50	38.8	78	21-133	
Nitrobenzene	ug/L	50	44.2	88	35-180	
Pentachlorophenol	ug/L	50	33.9J	68	14-176	
Phenanthrene	ug/L	50	48.5	97	54-120	
Phenol	ug/L	50	26.1	52	5-120	
Pyrene	ug/L	50	56.4	113	52-120	
2,4,6-Tribromophenol (S)	%			90	27-125	
2-Fluorobiphenyl (S)	%			64	32-92	
2-Fluorophenol (S)	%			55	9-74	
Nitrobenzene-d5 (S)	%			74	15-314	
p-Terphenyl-d14 (S)	%			94	8-146	
Phenol-d5 (S)	%			40	8-424	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch: 697392 Analysis Method: EPA 625.1  
QC Batch Method: EPA 625.1 Analysis Description: 625.1 MSS  
Laboratory: Pace Analytical Services - Indianapolis

Associated Lab Samples: 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

METHOD BLANK: 3206608 Matrix: Water  
Associated Lab Samples: 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
1,2,4-Trichlorobenzene	ug/L	ND	10.0	3.7	09/27/22 18:15	
1,2-Dichlorobenzene	ug/L	ND	10.0	2.7	09/27/22 18:15	
1,2-Diphenylhydrazine	ug/L	ND	10.0	2.1	09/27/22 18:15	N2
1,3-Dichlorobenzene	ug/L	ND	10.0	2.9	09/27/22 18:15	
1,4-Dichlorobenzene	ug/L	ND	10.0	2.9	09/27/22 18:15	
2,4,6-Trichlorophenol	ug/L	ND	10.0	2.8	09/27/22 18:15	
2,4-Dichlorophenol	ug/L	ND	10.0	2.3	09/27/22 18:15	
2,4-Dimethylphenol	ug/L	ND	10.0	2.3	09/27/22 18:15	
2,4-Dinitrophenol	ug/L	ND	50.0	5.2	09/27/22 18:15	
2,4-Dinitrotoluene	ug/L	ND	10.0	2.4	09/27/22 18:15	
2,6-Dinitrotoluene	ug/L	ND	10.0	2.9	09/27/22 18:15	
2-Chloronaphthalene	ug/L	ND	10.0	2.6	09/27/22 18:15	
2-Chlorophenol	ug/L	ND	10.0	2.3	09/27/22 18:15	
2-Nitrophenol	ug/L	ND	10.0	4.2	09/27/22 18:15	
3,3'-Dichlorobenzidine	ug/L	ND	20.0	3.1	09/27/22 18:15	
4,6-Dinitro-2-methylphenol	ug/L	ND	50.0	8.2	09/27/22 18:15	
4-Bromophenylphenyl ether	ug/L	ND	10.0	2.9	09/27/22 18:15	
4-Chloro-3-methylphenol	ug/L	ND	20.0	3.0	09/27/22 18:15	
4-Chlorophenylphenyl ether	ug/L	ND	10.0	2.7	09/27/22 18:15	
4-Nitrophenol	ug/L	ND	50.0	5.1	09/27/22 18:15	
Acenaphthene	ug/L	ND	10.0	1.8	09/27/22 18:15	
Acenaphthylene	ug/L	ND	10.0	1.9	09/27/22 18:15	
Anthracene	ug/L	ND	10.0	1.9	09/27/22 18:15	
Benzidine	ug/L	ND	50.0	6.0	09/27/22 18:15	
Benzo(a)anthracene	ug/L	ND	10.0	1.7	09/27/22 18:15	
Benzo(a)pyrene	ug/L	ND	10.0	2.3	09/27/22 18:15	
Benzo(b)fluoranthene	ug/L	ND	10.0	2.6	09/27/22 18:15	
Benzo(g,h,i)perylene	ug/L	ND	10.0	2.2	09/27/22 18:15	
Benzo(k)fluoranthene	ug/L	ND	10.0	2.6	09/27/22 18:15	
bis(2-Chloroethoxy)methane	ug/L	ND	10.0	3.1	09/27/22 18:15	
bis(2-Chloroethyl) ether	ug/L	ND	10.0	2.2	09/27/22 18:15	
bis(2-Chloroisopropyl) ether	ug/L	ND	10.0	3.6	09/27/22 18:15	
bis(2-Ethylhexyl)phthalate	ug/L	ND	5.0	4.2	09/27/22 18:15	
Butylbenzylphthalate	ug/L	ND	10.0	3.7	09/27/22 18:15	
Chrysene	ug/L	ND	10.0	1.9	09/27/22 18:15	
Di-n-butylphthalate	ug/L	ND	10.0	3.3	09/27/22 18:15	
Di-n-octylphthalate	ug/L	ND	10.0	7.3	09/27/22 18:15	
Dibenz(a,h)anthracene	ug/L	ND	10.0	2.6	09/27/22 18:15	
Diethylphthalate	ug/L	ND	10.0	3.2	09/27/22 18:15	
Dimethylphthalate	ug/L	ND	10.0	2.3	09/27/22 18:15	

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

METHOD BLANK: 3206608

Matrix: Water

Associated Lab Samples: 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Fluoranthene	ug/L	ND	10.0	1.8	09/27/22 18:15	
Fluorene	ug/L	ND	10.0	2.0	09/27/22 18:15	
Hexachloro-1,3-butadiene	ug/L	ND	10.0	3.8	09/27/22 18:15	
Hexachlorobenzene	ug/L	ND	10.0	3.3	09/27/22 18:15	
Hexachlorocyclopentadiene	ug/L	ND	20.0	4.5	09/27/22 18:15	N2
Hexachloroethane	ug/L	ND	10.0	3.4	09/27/22 18:15	N2
Indeno(1,2,3-cd)pyrene	ug/L	ND	10.0	2.6	09/27/22 18:15	
Isophorone	ug/L	ND	10.0	1.9	09/27/22 18:15	
N-Nitroso-di-n-propylamine	ug/L	ND	10.0	3.0	09/27/22 18:15	
N-Nitrosodimethylamine	ug/L	ND	20.0	3.5	09/27/22 18:15	
N-Nitrosodiphenylamine	ug/L	ND	10.0	2.2	09/27/22 18:15	
Naphthalene	ug/L	ND	10.0	2.4	09/27/22 18:15	
Nitrobenzene	ug/L	ND	10.0	3.1	09/27/22 18:15	
Pentachlorophenol	ug/L	ND	50.0	6.7	09/27/22 18:15	
Phenanthrene	ug/L	ND	10.0	1.9	09/27/22 18:15	
Phenol	ug/L	ND	10.0	1.2	09/27/22 18:15	
Pyrene	ug/L	ND	10.0	2.0	09/27/22 18:15	
2,4,6-Tribromophenol (S)	%	79	27-125		09/27/22 18:15	
2-Fluorobiphenyl (S)	%	64	32-92		09/27/22 18:15	
2-Fluorophenol (S)	%	46	9-74		09/27/22 18:15	
Nitrobenzene-d5 (S)	%	77	15-314		09/27/22 18:15	
p-Terphenyl-d14 (S)	%	87	8-146		09/27/22 18:15	
Phenol-d5 (S)	%	31	8-424		09/27/22 18:15	

LABORATORY CONTROL SAMPLE: 3206609

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
1,2,4-Trichlorobenzene	ug/L	50	36.6	73	44-142	
1,2-Dichlorobenzene	ug/L	50	30.3	61	31-79	
1,2-Diphenylhydrazine	ug/L	50	50.0	100	59-111	N2
1,3-Dichlorobenzene	ug/L	50	29.3	59	28-73	
1,4-Dichlorobenzene	ug/L	50	31.2	62	29-76	
2,4,6-Trichlorophenol	ug/L	50	42.9	86	37-144	
2,4-Dichlorophenol	ug/L	50	42.2	84	39-135	
2,4-Dimethylphenol	ug/L	50	42.7	85	32-120	
2,4-Dinitrophenol	ug/L	50	29.6J	59	1-191	
2,4-Dinitrotoluene	ug/L	50	46.0	92	39-139	
2,6-Dinitrotoluene	ug/L	50	47.3	95	50-158	
2-Chloronaphthalene	ug/L	50	43.5	87	60-120	
2-Chlorophenol	ug/L	50	36.6	73	23-134	
2-Nitrophenol	ug/L	50	41.2	82	29-182	
3,3'-Dichlorobenzidine	ug/L	100	88.7	89	1-262	
4,6-Dinitro-2-methylphenol	ug/L	50	39.9J	80	1-181	
4-Bromophenylphenyl ether	ug/L	50	46.6	93	53-127	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

**REPORT OF LABORATORY ANALYSIS**

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### QUALITY CONTROL DATA

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

LABORATORY CONTROL SAMPLE: 3206609

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
4-Chloro-3-methylphenol	ug/L	50	43.7	87	22-147	
4-Chlorophenylphenyl ether	ug/L	50	43.2	86	25-158	
4-Nitrophenol	ug/L	50	24.4J	49	1-132	
Acenaphthene	ug/L	50	45.5	91	47-145	
Acenaphthylene	ug/L	50	41.5	83	33-145	
Anthracene	ug/L	50	47.7	95	27-133	
Benzidine	ug/L	100	16.9J	17	1-64	
Benzo(a)anthracene	ug/L	50	49.9	100	33-143	
Benzo(a)pyrene	ug/L	50	45.1	90	17-163	
Benzo(b)fluoranthene	ug/L	50	49.4	99	24-159	
Benzo(g,h,i)perylene	ug/L	50	50.4	101	1-219	
Benzo(k)fluoranthene	ug/L	50	45.7	91	11-162	
bis(2-Chloroethoxy)methane	ug/L	50	46.5	93	33-184	
bis(2-Chloroethyl) ether	ug/L	50	40.8	82	12-158	
bis(2-Chloroisopropyl) ether	ug/L	50	41.3	83	36-166	
bis(2-Ethylhexyl)phthalate	ug/L	50	56.3	113	8-158	
Butylbenzylphthalate	ug/L	50	52.9	106	1-152	
Chrysene	ug/L	50	51.7	103	17-168	
Di-n-butylphthalate	ug/L	50	48.5	97	1-120	
Di-n-octylphthalate	ug/L	50	51.6	103	4-146	
Dibenz(a,h)anthracene	ug/L	50	51.1	102	1-227	
Diethylphthalate	ug/L	50	47.2	94	1-120	
Dimethylphthalate	ug/L	50	45.2	90	1-120	
Fluoranthene	ug/L	50	46.8	94	26-137	
Fluorene	ug/L	50	46.7	93	59-121	
Hexachloro-1,3-butadiene	ug/L	50	34.0	68	24-120	
Hexachlorobenzene	ug/L	50	47.2	94	1-152	
Hexachlorocyclopentadiene	ug/L	50	36.1	72	5-92 N2	
Hexachloroethane	ug/L	50	27.8	56	40-120 N2	
Indeno(1,2,3-cd)pyrene	ug/L	50	51.8	104	1-171	
Isophorone	ug/L	50	45.2	90	21-196	
N-Nitroso-di-n-propylamine	ug/L	50	40.8	82	1-230	
N-Nitrosodimethylamine	ug/L	50	25.8	52	1-107	
N-Nitrosodiphenylamine	ug/L	50	48.7	97	65-108	
Naphthalene	ug/L	50	37.9	76	21-133	
Nitrobenzene	ug/L	50	42.7	85	35-180	
Pentachlorophenol	ug/L	50	30.6J	61	14-176	
Phenanthrene	ug/L	50	48.9	98	54-120	
Phenol	ug/L	50	20.3	41	5-120	
Pyrene	ug/L	50	49.7	99	52-120	
2,4,6-Tribromophenol (S)	%			79	27-125	
2-Fluorobiphenyl (S)	%			63	32-92	
2-Fluorophenol (S)	%			47	9-74	
Nitrobenzene-d5 (S)	%			79	15-314	
p-Terphenyl-d14 (S)	%			90	8-146	
Phenol-d5 (S)	%			30	8-424	

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### REPORT OF LABORATORY ANALYSIS

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**QUALITY CONTROL DATA**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

QC Batch:	697620	Analysis Method:	EPA 335.4
QC Batch Method:	EPA 335.4	Analysis Description:	335.4 Cyanide, Total
		Laboratory:	Pace Analytical Services - Indianapolis

Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

METHOD BLANK: 3207750 Matrix: Water  
Associated Lab Samples: 50326308001, 50326308002, 50326308003, 50326308004, 50326308005, 50326308006, 50326308007, 50326308008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Cyanide	mg/L	ND	0.0050	0.0018	09/27/22 17:13	

LABORATORY CONTROL SAMPLE: 3207751

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Cyanide	mg/L	0.1	0.10	101	90-110	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3207752 3207753

Parameter	Units	50326606007 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Cyanide	mg/L	<0.0050	0.1	0.1	0.099	0.098	98	96	90-110	2	20	

MATRIX SPIKE SAMPLE: 3207754

Parameter	Units	50326308008 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Cyanide	mg/L	ND	0.1	0.10	98	90-110	

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**REPORT OF LABORATORY ANALYSIS**

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## QUALIFIERS

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

---

### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

TNTC - Too Numerous To Count

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit - The lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Reported results are not rounded until the final step prior to reporting. Therefore, calculated parameters that are typically reported as "Total" may vary slightly from the sum of the reported component parameters.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

### ANALYTE QUALIFIERS

H7 Re-extraction or re-analysis could not be performed within method holding time.

L1 Analyte recovery in the laboratory control sample (LCS) was above QC limits. Results for this analyte in associated samples may be biased high.

L2 Analyte recovery in the laboratory control sample (LCS) was below QC limits. Results for this analyte in associated samples may be biased low.

M0 Matrix spike recovery and/or matrix spike duplicate recovery was outside laboratory control limits.

M1 Matrix spike recovery exceeded QC limits. Batch accepted based on laboratory control sample (LCS) recovery.

N2 The lab does not hold NELAC/TNI accreditation for this parameter but other accreditations/certifications may apply. A complete list of accreditations/certifications is available upon request.

## REPORT OF LABORATORY ANALYSIS

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**QUALITY CONTROL DATA CROSS REFERENCE TABLE**

Project: THERMAL BIOASSAY STUDY  
Pace Project No.: 50326308

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
50326308001	20-CON-19SEP2022	EPA 608.3	697274	EPA 608.3	697762
50326308002	20-UPS-19SEP2022	EPA 608.3	697274	EPA 608.3	697762
50326308003	20-EFF-19SEP2022	EPA 608.3	697274	EPA 608.3	697762
50326308004	20-DNS-19SEP2022	EPA 608.3	697274	EPA 608.3	697762
50326308005	30-CON-19SEP2022	EPA 608.3	697274	EPA 608.3	697762
50326308006	30-UPS-19SEP2022	EPA 608.3	697274	EPA 608.3	697762
50326308007	30-EFF-19SEP2022	EPA 608.3	697778	EPA 608.3	697926
50326308008	30-DNS-19SEP2022	EPA 608.3	697778	EPA 608.3	697926
50326308001	20-CON-19SEP2022	EPA 608.3	697276	EPA 608.3	697763
50326308002	20-UPS-19SEP2022	EPA 608.3	697276	EPA 608.3	697763
50326308003	20-EFF-19SEP2022	EPA 608.3	697276	EPA 608.3	697763
50326308004	20-DNS-19SEP2022	EPA 608.3	697276	EPA 608.3	697763
50326308005	30-CON-19SEP2022	EPA 608.3	697276	EPA 608.3	697763
50326308006	30-UPS-19SEP2022	EPA 608.3	697276	EPA 608.3	697763
50326308007	30-EFF-19SEP2022	EPA 608.3	697779	EPA 608.3	697927
50326308008	30-DNS-19SEP2022	EPA 608.3	697779	EPA 608.3	697927
50326308001	20-CON-19SEP2022	EPA 200.8	697950	EPA 200.8	698179
50326308002	20-UPS-19SEP2022	EPA 200.8	697950	EPA 200.8	698179
50326308003	20-EFF-19SEP2022	EPA 200.8	697950	EPA 200.8	698179
50326308004	20-DNS-19SEP2022	EPA 200.8	697950	EPA 200.8	698179
50326308005	30-CON-19SEP2022	EPA 200.8	697950	EPA 200.8	698179
50326308006	30-UPS-19SEP2022	EPA 200.8	697950	EPA 200.8	698179
50326308007	30-EFF-19SEP2022	EPA 200.8	697950	EPA 200.8	698179
50326308008	30-DNS-19SEP2022	EPA 200.8	697950	EPA 200.8	698179
50326308001	20-CON-19SEP2022	EPA 245.1	697565	EPA 245.1	697811
50326308002	20-UPS-19SEP2022	EPA 245.1	698406	EPA 245.1	698732
50326308003	20-EFF-19SEP2022	EPA 245.1	698406	EPA 245.1	698732
50326308004	20-DNS-19SEP2022	EPA 245.1	698406	EPA 245.1	698732
50326308005	30-CON-19SEP2022	EPA 245.1	698406	EPA 245.1	698732
50326308006	30-UPS-19SEP2022	EPA 245.1	698406	EPA 245.1	698732
50326308007	30-EFF-19SEP2022	EPA 245.1	698406	EPA 245.1	698732
50326308008	30-DNS-19SEP2022	EPA 245.1	698406	EPA 245.1	698732
50326308001	20-CON-19SEP2022	EPA 625.1	697239	EPA 625.1	697457
50326308002	20-UPS-19SEP2022	EPA 625.1	697239	EPA 625.1	697457
50326308003	20-EFF-19SEP2022	EPA 625.1	697239	EPA 625.1	697457
50326308004	20-DNS-19SEP2022	EPA 625.1	697392	EPA 625.1	697962
50326308005	30-CON-19SEP2022	EPA 625.1	697392	EPA 625.1	697962
50326308006	30-UPS-19SEP2022	EPA 625.1	697392	EPA 625.1	697962
50326308007	30-EFF-19SEP2022	EPA 625.1	697392	EPA 625.1	697962
50326308008	30-DNS-19SEP2022	EPA 625.1	697392	EPA 625.1	697962
50326308001	20-CON-19SEP2022	EPA 624.1	696949		
50326308002	20-UPS-19SEP2022	EPA 624.1	696949		
50326308003	20-EFF-19SEP2022	EPA 624.1	696949		
50326308004	20-DNS-19SEP2022	EPA 624.1	696949		

**REPORT OF LABORATORY ANALYSIS**

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**QUALITY CONTROL DATA CROSS REFERENCE TABLE**

Project: THERMAL BIOASSAY STUDY

Pace Project No.: 50326308

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
50326308005	30-CON-19SEP2022	EPA 624.1	696949		
50326308006	30-UPS-19SEP2022	EPA 624.1	696949		
50326308007	30-EFF-19SEP2022	EPA 624.1	696949		
50326308008	30-DNS-19SEP2022	EPA 624.1	696949		
50326308001	20-CON-19SEP2022	EPA 335.4	697620	EPA 335.4	698000
50326308002	20-UPS-19SEP2022	EPA 335.4	697620	EPA 335.4	698000
50326308003	20-EFF-19SEP2022	EPA 335.4	697620	EPA 335.4	698000
50326308004	20-DNS-19SEP2022	EPA 335.4	697620	EPA 335.4	698000
50326308005	30-CON-19SEP2022	EPA 335.4	697620	EPA 335.4	698000
50326308006	30-UPS-19SEP2022	EPA 335.4	697620	EPA 335.4	698000
50326308007	30-EFF-19SEP2022	EPA 335.4	697620	EPA 335.4	698000
50326308008	30-DNS-19SEP2022	EPA 335.4	697620	EPA 335.4	698000

**REPORT OF LABORATORY ANALYSIS**

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**SAMPLE CONDITION UPON RECEIPT FORM**



Date/Time and Initials of person examining contents: 9/20/22 @ 1255 DF

1. Courier:  FED EX  UPS  CLIENT  PACE  USPS  OTHER Sett

5. Packing Material:  Bubble Wrap  Bubble Bags  
 None  Other \_\_\_\_\_

2. Custody Seal on Cooler/Box Present:  Yes  No  
(If yes)Seals Intact:  Yes  No (leave blank if no seals were present)

6. Ice Type:  Wet  Blue  None

3. Thermometer: 1 2 3 4 5 6 A B C D E F  
4. Cooler Temperature(s): 4.0 / 4.2 25/27 32/34 32/34 36/38 7.3/9.6 4.1/4.9

7. If temp. is over 6°C or under 0°C, was the PM notified?:  Yes  No  
Cooler temp should be above freezing to 6°C

(Initial/Corrected) RECORD TEMPS OF ALL COOLERS RECEIVED (use Comments below to add more) 28/30

All discrepancies will be written out in the comments section below.

	Yes	No		Yes	No	N/A
USDA Regulated Soils? (HI, ID, NY, WA, OR, CA, NM, TX, OK, AR, LA, TN, AL, MS, NC, SC, GA, FL, or Puerto Rico)		X	All containers needing acid/base preservation have been pH CHECKED?. Exceptions: VOA, coliform, LLHg, O&G, RAD CHEM, and any container with a septum cap or preserved with HCl.			
Short Hold Time Analysis (48 hours or less)? Analysis:		X	Circle: HNO3 (<2) H2SO4 (<2) NaOH (>10) NaOH/ZnAc (>9) Any non-conformance to pH recommendations will be noted on the container count form	X		
Time 5035A TC placed in Freezer or Short Holds To Lab	Time:		Residual Chlorine Check (SVOC 625 Pest/PCB 608)	Present	Absent	N/A
Rush TAT Requested (4 days or less): <u>3 DAY</u>	X		Residual Chlorine Check (Total/Amenable/Free Cyanide)		X	
Custody Signatures Present?		X	Headspace Wisconsin Sulfide?			X
Containers Intact?:	X		Headspace in VOA Vials (>6mm): See Container Count form for details	Present	Absent	No VOA Vials Sent
Sample Label (IDs/Dates/Times) Match COC?: Except TCs, which only require sample ID	X		Trip Blank Present?		X	
Extra labels on Terracore Vials? (soils only)		N/A	Trip Blank Custody Seals?:			X

COMMENTS:

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_





www.pacelabs.com

**Report Prepared for:**

Kenneth Hunt  
PACE Indianapolis  
7726 Moller Road  
Indianapolis IN 46268

**REPORT OF  
LABORATORY  
ANALYSIS FOR  
TCDD**

**Report Information:**

**PaceProject#: 10626458**  
**Sample Receipt Date: 09/21/2022**  
**Client Project #: 50326308 Marathon Rob**  
**Client Sub PO #: N/A**  
**State Cert #: 200011**

**Invoicing & Reporting Options:**

The report provided has been invoiced as a Level 2 2,3,7,8-TCDD Report. If an upgrade of this report package is requested, an additional charge may be applied.

Please review the attached invoice for accuracy and forward any questions to Carolynne Trout, your Pace Project Manager.

**This report has been reviewed by:**

September 30, 2022

Carolynne Trout, Project Manager  
(612) 607-6351  
(612) 607-6444 (fax)  
Carolynne.Trout@pacelabs.com



**Report of Laboratory Analysis**

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The results relate only to the samples included in this report.

**Report Prepared Date:**

September 29, 2022



## **DISCUSSION**

This report presents the results from the analyses performed on eight samples submitted by a representative of Pace Analytical Services, LLC. The samples were analyzed for the presence or absence of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) using USEPA Method 1613B. The reporting limits were set to correspond to the lowest calibration point and a nominal 1-Liter sample amount, and the sensitivity was verified by signal-to-noise measurements. The quantitation limits, adjusted for sample extraction amount, may be somewhat higher or lower than the reporting limits provided in this report.

The recoveries of the isotopically-labeled TCDD internal standard in the sample extracts ranged from 52-74%. All of the labeled standard recoveries obtained for this project were within the target ranges specified in Method 1613B. Also, since the quantification of the native TCDD was based on isotope dilution, the data were automatically corrected for recovery and accurate values were obtained.

A laboratory method blank was prepared and analyzed with the sample batch as part of our routine quality control procedures. The results show the blank to be free of 2,3,7,8-TCDD at the reporting limit.

Laboratory spike samples were also prepared using clean reference matrix that had been fortified with native standard material. The results show that the spiked native TCDD was recovered at 97-100% with a relative percent difference of 3.0%. These results were within the target ranges for the method. Matrix spikes were not prepared with the sample batch.

## **REPORT OF LABORATORY ANALYSIS**

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## Minnesota Laboratory Certifications

Authority	Certificate #	Authority	Certificate #
A2LA	2926.01	Mississippi	MN00064
Alabama	40770	Missouri	10100
Alaska-DW	MN00064	Montana	CERT0092
Alaska-UST	17-009	Nebraska	NE-OS-18-06
Arizona	AZ0014	Nevada	MN00064
Arkansas - WW	88-0680	New Hampshire	2081
Arkansas-DW	MN00064	New Jersey	MN002
California	2929	New York	11647
Colorado	MN00064	North Carolina-	27700
Connecticut	PH-0256	North Carolina-	530
Florida	E87605	North Dakota	R-036
Georgia	959	Ohio-DW	41244
Hawaii	MN00064	Ohio-VAP (170	CL101
Idaho	MN00064	Ohio-VAP (180	CL110
Illinois	200011	Oklahoma	9507
Indiana	C-MN-01	Oregon- rimary	MN300001
Iowa	368	Oregon-Second	MN200001
Kansas	E-10167	Pennsylvania	68-00563
Kentucky-DW	90062	Puerto Rico	MN00064
Kentucky-WW	90062	South Carolina	74003
Louisiana-DEQ	AI-84596	Tennessee	TN02818
Louisiana-DW	MN00064	Texas	T104704192
Maine	MN00064	Utah	MN00064
Maryland	322	Vermont	VT-027053137
Michigan	9909	Virginia	460163
Minnesota	027-053-137	Washington	C486
Minnesota-Ag	via MN 027-053	West Virginia-D	382
Minnesota-Petr	1240	West Virginia-D	9952C
		Wisconsin	999407970
		Wyoming-UST	via A2LA 2926.

## REPORT OF LABORATORY ANALYSIS

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Report No.....10626458

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**Pace Analytical Services, LLC**  
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Minneapolis, MN 55414  
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Fax: 612.607.6444  
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## **Appendix A**

### **Sample Management**

## **REPORT OF LABORATORY ANALYSIS**

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# Internal Transfer Chain of Custody



Samples Pre-Logged into eCOC.

State of Origin: IL  
 Cert. Needed:  Yes  No

Owner Received Date: 9/20/2022 Results Requested By: 10/11/2022



Workorder: 50326308 Report To: **Thermal Bioassay Study**

Kenneth Hunt  
 Pace Analytical Indianapolis  
 7726 Moller Road  
 Indianapolis, IN 46268  
 Phone (317)228-3100

Pace Analytical Minnesota  
 1700 Elm Street  
 Suite 200  
 Minneapolis, MN 55414  
 Phone (612)607-1700

WO#: 10626458

Transfers	Released By	Date/Time	Received By	Date/Time	Received on Ice	Y or N	Samples Intact	Y or N
1	20-CON-19SEP2022	9/19/2022 12:45	50326308001	Water	2	X		001
2	20-UPS-19SEP2022	9/19/2022 16:35	50326308002	Water	2	X		002
3	20-EFF-19SEP2022	9/19/2022 15:35	50326308003	Water	2	X		003
4	20-DNS-19SEP2022	9/19/2022 12:10	50326308004	Water	2	X		004
5	30-CON-19SEP2022	9/19/2022 16:05	50326308005	Water	2	X		005
6	30-UPS-19SEP2022	9/19/2022 13:30	50326308006	Water	2	X		006
7	30-EFF-19SEP2022	9/19/2022 16:20	50326308007	Water	2	X		007
8	30-DNS-19SEP2022	9/19/2022 15:50	50326308008	Water	2	X		008

Transfers Released By: *[Signature]* Date/Time: 9/20/22 17:30  
 Received By: *[Signature]* Date/Time: 9/21/22 18:40

Cooler Temperature on Receipt: 4.34 °C Custody Seal: Y or N

\*\*\*In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document. This chain of custody is considered complete as is since this information is available in the owner laboratory.

Sample Condition Upon Receipt  
 Client Name: Pace Monitoring

Project #: **WO# : 10626458**  
 PM: CT1 Due Date: 09/28/22  
 CLIENT: PASI-INDI

Courier:  FedEx  UPS  USPS  Client  
 Pace  Speedee  Commercial

Tracking Number: 5849 Usas 8710  See Exceptions ENV-FRM-MIN4-0142

Custody Seal on Cooler/Box Present?  Yes  No Seals Intact?  Yes  No Biological Tissue Frozen?  Yes  No  N/A  
 Packing Material:  Bubble Wrap  Bubble Bags  None  Other Temp Blank?  Yes  No  
 Thermometer:  T1 (0461)  T2 (1336)  T3 (0459)  T4 (0254)  T5 (0178)  
 T6 (0235)  T7 (0042)  T8 (0775)  01339252/1710 Type of Ice:  Wet  Blue  Dry  None  
 Melted

Did Samples Originate in West Virginia?  Yes  No Were All Container Temps Taken?  Yes  No  N/A  
 Temp should be above freezing to 6 °C Cooler temp Read w/Temp Blank: 3.448 °C Average Corrected Temp (no temp blank only): \_\_\_\_\_ °C  
 Correction Factor: None Cooler Temp Corrected w/temp blank: 3.448  See Exceptions ENV-FRM-MIN4-0142  1 Container

USDA Regulated Soil:  N/A, water sample/other: \_\_\_\_\_ Date/Initials of Person Examining Contents: 9/22/22  
 Did samples originate in a quarantine zone within the United States: AL, AR, AZ CA, FL, GA, ID, LA, MS, NC, NM, NY, OK, OR, SC, TN, TX, or VA (check maps)?  Yes  No  
 Did samples originate from a foreign source (internationally, including Hawaii and Puerto Rico)?  Yes  No

If Yes to either question, fill out a Regulated Soil Checklist (ENV-FRM-MIN4-0154) and include with SCUR/COC paperwork.

Location (Check one): <input type="checkbox"/> Duluth <input checked="" type="checkbox"/> Minneapolis <input type="checkbox"/> Virginia	COMMENTS
Chain of Custody Present and Filled Out? <input checked="" type="checkbox"/> Yes <input checked="" type="checkbox"/> No	1.
Chain of Custody Relinquished? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	2.
Sampler Name and/or Signature on COC? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	3.
Samples Arrived within Hold Time? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	4. If fecal: <input type="checkbox"/> <8 hrs <input type="checkbox"/> >8 hr, <24 <input type="checkbox"/> No
Short Hold Time Analysis (<72 hr)? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	5. <input type="checkbox"/> Fecal Coliform <input type="checkbox"/> HPC <input type="checkbox"/> Total Coliform/E.coli <input type="checkbox"/> BOD/cBOD <input type="checkbox"/> Hex Chrom <input type="checkbox"/> Turbidity <input type="checkbox"/> Nitrate <input type="checkbox"/> Nitrite <input type="checkbox"/> Orthophos <input type="checkbox"/> Other
Rush Turn Around Time Requested? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	6.
Sufficient Sample Volume? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	7.
Correct Containers Used? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8.
-Pace Containers Used? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	
Containers Intact? <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No	9.
Field Filtered Volume Received for Dissolved Tests? <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	10. Is sediment visible in the dissolved container? <input type="checkbox"/> Yes <input type="checkbox"/> No
Is sufficient information available to reconcile the samples to the COC? Matrix: <input checked="" type="checkbox"/> Water <input type="checkbox"/> Soil <input type="checkbox"/> Oil <input type="checkbox"/> Other	11. If no, write ID/Date/Time of container below: <input type="checkbox"/> See Exceptions ENV-FRM-MIN4-0142
All containers needing acid/base preservation have been checked? <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	12. Sample # <input type="checkbox"/> NaOH <input type="checkbox"/> HNO3 <input type="checkbox"/> H2SO4 <input type="checkbox"/> Zinc Acetate
All containers needing preservation are found to be in compliance with EPA recommendation (HNO3, H2SO4, <2pH, NaOH >9 Sulfide, NaOH >10 Cyanide) Exceptions: VOA, Coliform, TOC/DOC Oil and Grease, DRO/8015 (water) and Dioxins/PFAS (*If adding preservative to a container, it must be added to associated field and equipment blanks--verify with PM first.)	Positive for Residual Chlorine? <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> See Exceptions ENV-FRM-MIN4-0142 pH Paper Lot # Residual Chlorine 0-6 Roll 0-6 Strip 0-14 Strip
Headspace in Methyl Mercury Container? <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	13.
Extra labels present on soil VOA or WIDRO containers? <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	14. <input type="checkbox"/> See Exceptions ENV-FRM-MIN4-0142
Headspace in VOA Vials (greater than 6mm)? <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	
3 Trip Blanks Present? <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	15.
Trip Blank Custody Seals Present? <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	Pace Trip Blank Lot # (if purchased): _____

CLIENT NOTIFICATION/RESOLUTION  
 Person Contacted: \_\_\_\_\_ Date/Time: \_\_\_\_\_  
 Comments/Resolution: \_\_\_\_\_  
 Project Manager Review: Carolynne Trout Date: 9/22/22  
 Field Data Required?  Yes  No

NOTE: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHR Certification Office (i.e., out of hold, incorrect preservative, out of temp, incorrect containers).  
 Labeled By: ME Line: (Signature)



F-IN-Q-290-rev.22, 22Apr2022

**SAMPLE CONDITION UPON RECEIPT FORM**

*Pace*

Date/Time and Initials of person examining contents: 9/20/20 DLSS DF

1. Courier:  FED EX  UPS  CLIENT  PACE  USPS  OTHER Self
2. Custody Seal on Cooler/Box Present:  Yes  No (leave blank if no seals were present)
3. Thermometer: 1 2 3 4 5 6 A B C D E F
4. Cooler Temperature(s): 4.0 / 4.2 / 25/27 / 29/31 / 32/34 / 36/38 / 37/35 / 41/43
5. Packing Material:  Bubble Wrap  Bubble Bags  
 None  Other
6. Ice Type:  Wet  Blue  None  
 Dry if temp. is over 6°C or under 0°C, was the PM notified?:  Yes  No
7. Cooler temp should be above freezing to 6°C

All discrepancies will be written out in the comments section below.

RECORD TEMPS OF ALL COOLERS RECEIVED (use Comments below to add more)		Yes	No	Yes	No	N/A
USDA Regulated Soils? (HI, ID, NY, WA, OR, CA, NM, TX, OK, AR, LA, TN, AL, MS, NC, SC, GA, FL, or Puerto Rico)			X			
Short Hold Time Analysis (48 hours or less)? Analysis: <u>NO3</u> ( <u>2</u> ) <u>H2SO4</u> ( <u>2</u> ) <u>NaOH</u> ( <u>&gt;10</u> ) <u>NaOH/ZnAc</u> ( <u>&gt;9</u> ) <i>Why not conformance to pH recommendations will be noted on the container count form</i>			X	X		
Time 5035A TC placed in Freezer or Short Holds To Lab Time:				Present	Absent	N/A
Rush TAT Requested (4 days or less): <u>3 DAY</u>		X			X	
Custody Signatures Present?			X			X
Containers Intact?		X		Present	Absent	No VOA Vials Sent
Sample Label (ID's/Dates/Times) Match COC? Except TCs, which only require sample ID		X				X
Extra labels on Terracore Vials? (soils only)					X	X

COMMENTS:

Sample Container Count

\*\* Place a RED dot on containers that are out of conformance \*\*

COC Line Item	WGFU	Tech Cont./SBS	DI	VIALS		AMBER GLASS					PLASTIC					OTHER		Nitric Red	Sulfuric Yellow	Sodium Hydroxide Green	Sodium Hydroxide/ZnAc Black
				VOA VIAL HS (8-8mm)	DG9H VG9H	DG9U VG9U	AG1U AG1H	AG2U AG3S	AG3SF AG3C	BP1U BP1N	BP2U BP3U	BP3F BP3S	BP3B BP3Z	CG3H Syringe Kit							
1																					
2																					
3																					
4																					
5																					
6																					
7																					
8																					
9																					
10																					
11																					
12																					

Glass		Plastic	
G9H 40mL HCl amber vial	BG1T 1L Na Thiosulfate clear glass	BP1B 1L NaOH plastic	BP4U 125mL unpreserved plastic
G9P 40mL TSP amber vial	BG1U 1L unpreserved glass	BP1N 1L HNO3 plastic	BP4N 125mL HNO3 plastic
G9S 40mL H2SO4 amber vial	BG3H 250mL HCl Clear Glass	BP1S 1L H2SO4 plastic	BP4S 125mL H2SO4 plastic
G9T 40mL Na Thio amber vial	BG3U 250mL Unpres Clear Glass	BP1U 1L unpreserved plastic	
G9U 40mL unpreserved amber vial	AG0U 100mL unpres amber glass	BP1Z 1L NaOH, Zn, Ac	
G9H 40mL HCl clear vial	AG1H 1L HCl amber glass	BP2N 500mL HNO3 plastic	
G9T 40mL Na Thio. clear vial	AG1S 1L H2SO4 amber glass	BP2C 500mL NaOH plastic	
G9U 40mL unpreserved clear vial	AG1T 1L Na Thiosulfate amber glass	BP2S 500mL H2SO4 plastic	
I 40mL w/hexane wipe vial	AG1U 1liter unpres amber glass	BP2U 500mL unpreserved plastic	
GKU 8oz unpreserved clear jar	AG2N 500mL HNO3 amber glass	BP2Z 500mL NaOH, Zn, Ac	
GFU 4oz clear soil jar	AG2S 500mL H2SO4 amber glass	BP3B 250mL NaOH plastic	
BFU 4oz unpreserved amber wide	AG2U 500mL unpres amber glass	BP3N 250mL HNO3 plastic	
G3H 250mL clear glass HCl	AG3S 250mL H2SO4 amber glass	BP3F 250mL HNO3 plastic-field filtered	
B1H 1L HCl clear glass	AG3SF 250mL H2SO4 amb glass -field filtered	BP3U 250mL unpreserved plastic	
B1S 1L H2SO4 clear glass	AG3U 250mL unpres amber glass	BP3S 250mL H2SO4 plastic	
N General	AG3C 250mL NaOH amber glass	BP3Z 250mL NaOH, Zn, Ac plastic	

Miscellaneous

Syringe Kit	LL Cr+6 sampling kit
ZPLC	Ziploc Bag
R	Terracore Kit
SP5T	120mL Coliform Sodium Thiosulfate
T	Fedlar Bag (air sample)
U	Summa Can (air sample)
WT	Water
SL	Solid Solid
OL	Oil
NAL	Non-aqueous liquid
WP	Wipe

Container Codes



## Reporting Flags

- A = Reporting Limit based on signal to noise (EDL)
- B = Less than 10x higher than method blank level
- C = Result obtained from confirmation analysis
- D = Result obtained from analysis of diluted sample
- E = Exceeds calibration range
- I = Isotope ratio out of specification
- J = Estimated value
- L = Suppressive interference, analyte may be biased low
- Nn = Value obtained from additional analysis
- P = PCDE Interference
- R = Recovery outside target range
- S = Peak saturated
- U = Analyte not detected
- V = Result verified by confirmation analysis
- X = %D Exceeds limits
- Y = Calculated using average of daily RFs
- \* = See Discussion

## REPORT OF LABORATORY ANALYSIS

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## **Appendix B**

### **Sample Analysis Summary**

## **REPORT OF LABORATORY ANALYSIS**

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	20-CON-19SEP2022		
Lab Sample ID	50326308001		
Filename	L220928B_03		
Injected By	SMT		
Total Amount Extracted	995 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	09/19/2022 12:45
ICAL ID	L220811	Received	09/21/2022 08:40
CCal Filename(s)	L220928A_18	Extracted	09/23/2022 10:00
Method Blank ID	BLANK-101454	Analyzed	09/28/2022 23:33

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	58
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	72

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	20-UPS-19SEP2022		
Lab Sample ID	50326308002		
Filename	L220928B_04		
Injected By	SMT		
Total Amount Extracted	1000 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	09/19/2022 16:35
ICAL ID	L220811	Received	09/21/2022 08:40
CCal Filename(s)	L220928A_18	Extracted	09/23/2022 10:00
Method Blank ID	BLANK-101454	Analyzed	09/29/2022 00:16

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	53
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	76

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	20-EFF-19SEP2022		
Lab Sample ID	50326308003		
Filename	L220928B_05		
Injected By	SMT		
Total Amount Extracted	992 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	09/19/2022 15:35
ICAL ID	L220811	Received	09/21/2022 08:40
CCal Filename(s)	L220928A_18	Extracted	09/23/2022 10:00
Method Blank ID	BLANK-101454	Analyzed	09/29/2022 00:59

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	66
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	75

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	20-DNS-19SEP2022		
Lab Sample ID	50326308004		
Filename	L220928B_06		
Injected By	SMT		
Total Amount Extracted	970 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	09/19/2022 12:10
ICAL ID	L220811	Received	09/21/2022 08:40
CCal Filename(s)	L220928A_18	Extracted	09/23/2022 10:00
Method Blank ID	BLANK-101454	Analyzed	09/29/2022 01:42

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	61
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	71

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	30-CON-19SEP2022		
Lab Sample ID	50326308005		
Filename	L220928B_07		
Injected By	SMT		
Total Amount Extracted	983 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	09/19/2022 16:05
ICAL ID	L220811	Received	09/21/2022 08:40
CCal Filename(s)	L220928A_18	Extracted	09/23/2022 10:00
Method Blank ID	BLANK-101454	Analyzed	09/29/2022 02:25

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	52
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	63

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	30-UPS-19SEP2022		
Lab Sample ID	50326308006		
Filename	L220928B_08		
Injected By	SMT		
Total Amount Extracted	996 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	09/19/2022 13:30
ICAL ID	L220811	Received	09/21/2022 08:40
CCal Filename(s)	L220928A_18	Extracted	09/23/2022 10:00
Method Blank ID	BLANK-101454	Analyzed	09/29/2022 03:08

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	52
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	65

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	30-EFF-19SEP2022		
Lab Sample ID	50326308007		
Filename	L220928B_09		
Injected By	SMT		
Total Amount Extracted	1010 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	09/19/2022 16:20
ICAL ID	L220811	Received	09/21/2022 08:40
CCal Filename(s)	L220928A_18	Extracted	09/23/2022 10:00
Method Blank ID	BLANK-101454	Analyzed	09/29/2022 03:51

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	62
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	75

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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 Fax: 612-607-6444

**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	30-DNS-19SEP2022		
Lab Sample ID	50326308008		
Filename	L220928B_10		
Injected By	SMT		
Total Amount Extracted	973 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	09/19/2022 15:50
ICAL ID	L220811	Received	09/21/2022 08:40
CCal Filename(s)	L220928A_18	Extracted	09/23/2022 10:00
Method Blank ID	BLANK-101454	Analyzed	09/29/2022 04:34

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	74
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	87

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Blank Analysis Results**

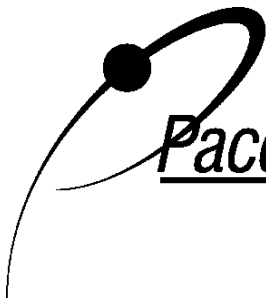
Lab Sample Name	DFBLKIZ	Matrix	Water
Lab Sample ID	BLANK-101454	Dilution	NA
Filename	L220928A_08	Extracted	09/23/2022 10:00
Total Amount Extracted	983 mL	Analyzed	09/28/2022 14:14
ICAL ID	L220811	Injected By	SMT
CCal Filename(s)	L220928A_01		

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	—	10	2,3,7,8-TCDD-13C	2.00	46
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	65

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

**REPORT OF LABORATORY ANALYSIS**

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**Method 1613B Laboratory Control Spike Results**

Lab Sample ID	LCS-101455	Matrix	Water
Filename	L220928A_02	Dilution	NA
Total Amount Extracted	992 mL	Extracted	09/23/2022 10:00
ICAL ID	L220811	Analyzed	09/28/2022 09:56
CCal Filename	L220928A_01	Injected By	SMT
Method Blank ID	BLANK-101454		

Compound	Cs	Cr	Lower Limit	Upper Limit	% Rec.
2,3,7,8-TCDD	10	9.7	7.3	14.6	97
2,3,7,8-TCDD-37Cl4	10	5.7	3.7	15.8	57
2,3,7,8-TCDD-13C	100	46	25.0	141.0	46

Cs = Concentration Spiked (ng/mL)  
 Cr = Concentration Recovered (ng/mL)  
 Rec. = Recovery (Expressed as Percent)  
 Control Limit Reference: Method 1613, Table 6, 10/94 Revision  
 R = Recovery outside of control limits  
 Nn = Value obtained from additional analysis  
 \* = See Discussion

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**Method 1613B Laboratory Control Spike Results**

Lab Sample ID	LCSD-101456	Matrix	Water
Filename	L220928A_03	Dilution	NA
Total Amount Extracted	971 mL	Extracted	09/23/2022 10:00
ICAL ID	L220811	Analyzed	09/28/2022 10:39
CCal Filename	L220928A_01	Injected By	SMT
Method Blank ID	BLANK-101454		

Compound	Cs	Cr	Lower Limit	Upper Limit	% Rec.
2,3,7,8-TCDD	10	10	7.3	14.6	100
2,3,7,8-TCDD-37Cl4	10	7.4	3.7	15.8	74
2,3,7,8-TCDD-13C	100	56	25.0	141.0	56

Cs = Concentration Spiked (ng/mL)  
 Cr = Concentration Recovered (ng/mL)  
 Rec. = Recovery (Expressed as Percent)  
 Control Limit Reference: Method 1613, Table 6, 10/94 Revision  
 R = Recovery outside of control limits  
 Nn = Value obtained from additional analysis  
 \* = See Discussion

**REPORT OF LABORATORY ANALYSIS**

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**Method 1613B**

**Spike Recovery Relative Percent Difference (RPD) Results**

Client PACE Indianapolis

Spike 1 ID LCS-101455  
Spike 1 Filename L220928A\_02

Spike 2 ID LCSD-101456  
Spike 2 Filename L220928A\_03

<b>Compound</b>	<b>Spike 1 %REC</b>	<b>Spike 2 %REC</b>	<b>%RPD</b>
2,3,7,8-TCDD	97	100	3.0

%REC = Percent Recovered

RPD = The difference between the two values divided by the mean value

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## **Appendix C**

### **Water Chemistry Laboratory Report 17 October 2022**

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November 01, 2022

Ms. Julie Holscher  
Marathon Petroleum Company (Robinson IL)  
100 Marathon Ave.  
Robinson, IL 62454

RE: Project: Thermal Bioassay Study  
Pace Project No.: 50328663

Dear Ms. Holscher:

Enclosed are the analytical results for sample(s) received by the laboratory on October 18, 2022. The results relate only to the samples included in this report. Results reported herein conform to the applicable TNI/NELAC Standards and the laboratory's Quality Manual, where applicable, unless otherwise noted in the body of the report.

The test results provided in this final report were generated by each of the following laboratories within the Pace Network:

- Pace Analytical Services - Indianapolis

If you have any questions concerning this report, please feel free to contact me.

Sincerely,



Kenneth Hunt  
kenneth.hunt@pacelabs.com  
(317)228-3100  
Project Manager

Enclosures

cc: Mr. Patrick Beabout, Marathon Robinson Refinery  
Ms. Sara Clough, Marathon Robinson Refinery  
Mr. Michael Elliott, Marathon Robinson  
Ms. Emily Gullett, Marathon Robinson Refinery  
Mr. Douglas McNary, Marathon Petroleum (Robinson IL)  
Mr. Dillon O'Kelly, Marathon Robinson  
Jared Ridge, Marathon Robinson



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**CERTIFICATIONS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

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**Pace Analytical Services Indianapolis**

7726 Moller Road, Indianapolis, IN 46268

Illinois Accreditation #: 200074

Indiana Drinking Water Laboratory #: C-49-06

Kansas/TNI Certification #: E-10177

Kentucky UST Agency Interest #: 80226

Kentucky WW Laboratory ID #: 98019

Michigan Drinking Water Laboratory #9050

Ohio VAP Certified Laboratory #: CL0065

Oklahoma Laboratory #: 9204

Texas Certification #: T104704355

Wisconsin Laboratory #: 999788130

USDA Soil Permit #: P330-19-00257

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**REPORT OF LABORATORY ANALYSIS**

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### SAMPLE SUMMARY

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Lab ID	Sample ID	Matrix	Date Collected	Date Received
50328663001	20-CON-10172022	Water	10/17/22 10:55	10/18/22 12:20
50328663002	20-UPS-10172022	Water	10/17/22 10:40	10/18/22 12:20
50328663003	20-EFF-10172022	Water	10/17/22 10:20	10/18/22 12:20
50328663004	20-DNS-10172022	Water	10/17/22 09:50	10/18/22 12:20
50328663005	30-CON-10172022	Water	10/17/22 12:45	10/18/22 12:20
50328663006	30-UPS-10172022	Water	10/17/22 12:25	10/18/22 12:20
50328663007	30-EFF-10172022	Water	10/17/22 12:05	10/18/22 12:20
50328663008	30-DNS-10172022	Water	10/17/22 11:35	10/18/22 12:20

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**SAMPLE ANALYTE COUNT**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
50328663001	20-CON-10172022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	KLP	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50328663002	20-UPS-10172022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	KLP	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50328663003	20-EFF-10172022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	KLP	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50328663004	20-DNS-10172022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	KLP	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50328663005	30-CON-10172022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	KLP	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50328663006	30-UPS-10172022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I

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**SAMPLE ANALYTE COUNT**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
50328663007	30-EFF-10172022	EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	KLP	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
		EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	KLP	32	PASI-I
		EPA 335.4	ZM	1	PASI-I
50328663008	30-DNS-10172022	EPA 608.3	KAV	8	PASI-I
		EPA 608.3	KAV	19	PASI-I
		EPA 200.8	CAW	12	PASI-I
		EPA 245.1	ILP	1	PASI-I
		EPA 625.1	JCM	63	PASI-I
		EPA 624.1	KLP	32	PASI-I
		EPA 335.4	ZM	1	PASI-I

PASI-I = Pace Analytical Services - Indianapolis

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 20-CON-10172022**      **Lab ID: 50328663001**      Collected: 10/17/22 10:55      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**608.3 PCB**      Analytical Method: EPA 608.3      Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

PCB-1016 (Aroclor 1016)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:08	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:08	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:08	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:08	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:08	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:08	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.096	0.049	1	10/19/22 18:18	10/25/22 16:08	11096-82-5	

**Surrogates**  
Tetrachloro-m-xylene (S)      69      %      1-123      1      10/19/22 18:18      10/25/22 16:08      877-09-8

**608.3 Pesticides**      Analytical Method: EPA 608.3      Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

Aldrin	ND	ug/L	0.048	0.0087	1	10/19/22 18:18	10/31/22 15:47	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 15:47	319-84-6	
beta-BHC	ND	ug/L	0.048	0.013	1	10/19/22 18:18	10/31/22 15:47	319-85-7	
delta-BHC	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 15:47	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 15:47	58-89-9	
Chlordane (Technical)	ND	ug/L	0.48	0.26	1	10/19/22 18:18	10/31/22 15:47	57-74-9	
4,4'-DDD	ND	ug/L	0.096	0.023	1	10/19/22 18:18	10/31/22 15:47	72-54-8	
4,4'-DDE	ND	ug/L	0.096	0.017	1	10/19/22 18:18	10/31/22 15:47	72-55-9	
4,4'-DDT	ND	ug/L	0.096	0.035	1	10/19/22 18:18	10/31/22 15:47	50-29-3	
Dieldrin	ND	ug/L	0.096	0.021	1	10/19/22 18:18	10/31/22 15:47	60-57-1	
Endosulfan I	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 15:47	959-98-8	
Endosulfan II	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 15:47	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.096	0.020	1	10/19/22 18:18	10/31/22 15:47	1031-07-8	
Endrin	ND	ug/L	0.096	0.026	1	10/19/22 18:18	10/31/22 15:47	72-20-8	
Endrin aldehyde	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 15:47	7421-93-4	
Heptachlor	ND	ug/L	0.048	0.0096	1	10/19/22 18:18	10/31/22 15:47	76-44-8	
Heptachlor epoxide	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 15:47	1024-57-3	
Toxaphene	ND	ug/L	0.96	0.35	1	10/19/22 18:18	10/31/22 15:47	8001-35-2	

**Surrogates**  
Decachlorobiphenyl (S)      61      %      1-140      1      10/19/22 18:18      10/31/22 15:47      2051-24-3

**200.8 Metals, Total ICMS**      Analytical Method: EPA 200.8      Preparation Method: EPA 200.8  
Pace Analytical Services - Indianapolis

Antimony	<b>0.00013J</b>	mg/L	0.0010	0.00013	1	10/19/22 08:00	10/20/22 13:27	7440-36-0	
Arsenic	<b>0.00043J</b>	mg/L	0.0010	0.00011	1	10/19/22 08:00	10/20/22 13:27	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	10/19/22 08:00	10/20/22 13:27	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	10/19/22 08:00	10/20/22 13:27	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	10/19/22 08:00	10/20/22 13:27	7440-47-3	
Copper	<b>0.0049</b>	mg/L	0.0010	0.00037	1	10/19/22 08:00	10/20/22 13:27	7440-50-8	
Lead	ND	mg/L	0.0010	0.000080	1	10/19/22 08:00	10/20/22 13:27	7439-92-1	
Nickel	<b>0.0022</b>	mg/L	0.00050	0.00039	1	10/19/22 08:00	10/20/22 13:27	7440-02-0	
Selenium	<b>0.0033</b>	mg/L	0.0010	0.00035	1	10/19/22 08:00	10/20/22 13:27	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	10/19/22 08:00	10/20/22 13:27	7440-22-4	

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### ANALYTICAL RESULTS

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 20-CON-10172022 Lab ID: 50328663001 Collected: 10/17/22 10:55 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	ND	mg/L	0.0010	0.000073	1	10/19/22 08:00	10/20/22 13:27	7440-28-0	
Zinc	0.038	mg/L	0.0030	0.0010	1	10/19/22 08:00	10/20/22 13:27	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/23/22 19:34	10/24/22 11:41	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.6	1.7	1	10/20/22 15:49	10/28/22 20:26	83-32-9	
Acenaphthylene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 20:26	208-96-8	
Anthracene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 20:26	120-12-7	
Benzdine	ND	ug/L	48.1	5.8	1	10/20/22 15:49	10/28/22 20:26	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.6	1.6	1	10/20/22 15:49	10/28/22 20:26	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 20:26	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 20:26	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 20:26	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 20:26	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 20:26	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.6	3.6	1	10/20/22 15:49	10/28/22 20:26	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.2	2.9	1	10/20/22 15:49	10/28/22 20:26	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 20:26	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 20:26	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 20:26	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 20:26	91-58-7	
2-Chlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 20:26	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 20:26	7005-72-3	
Chrysene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 20:26	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 20:26	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 20:26	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 20:26	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 20:26	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.2	3.0	1	10/20/22 15:49	10/28/22 20:26	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 20:26	120-83-2	
Diethylphthalate	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 20:26	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 20:26	105-67-9	
Dimethylphthalate	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 20:26	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 20:26	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	48.1	7.8	1	10/20/22 15:49	10/28/22 20:26	534-52-1	
2,4-Dinitrophenol	ND	ug/L	48.1	5.0	1	10/20/22 15:49	10/28/22 20:26	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 20:26	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 20:26	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.6	7.1	1	10/20/22 15:49	10/28/22 20:26	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.6	2.0	1	10/20/22 15:49	10/28/22 20:26	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.8	4.0	1	10/20/22 15:49	10/28/22 20:26	117-81-7	

### REPORT OF LABORATORY ANALYSIS

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### ANALYTICAL RESULTS

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 20-CON-10172022 Lab ID: 50328663001 Collected: 10/17/22 10:55 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 20:26	206-44-0	
Fluorene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 20:26	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.6	3.7	1	10/20/22 15:49	10/28/22 20:26	87-68-3	
Hexachlorobenzene	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 20:26	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.2	4.4	1	10/20/22 15:49	10/28/22 20:26	77-47-4	N2
Hexachloroethane	ND	ug/L	9.6	3.3	1	10/20/22 15:49	10/28/22 20:26	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 20:26	193-39-5	
Isophorone	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 20:26	78-59-1	
Naphthalene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 20:26	91-20-3	
Nitrobenzene	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 20:26	98-95-3	
2-Nitrophenol	ND	ug/L	9.6	4.0	1	10/20/22 15:49	10/28/22 20:26	88-75-5	
4-Nitrophenol	ND	ug/L	48.1	4.9	1	10/20/22 15:49	10/28/22 20:26	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.2	3.4	1	10/20/22 15:49	10/28/22 20:26	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 20:26	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 20:26	86-30-6	
Pentachlorophenol	ND	ug/L	48.1	6.5	1	10/20/22 15:49	10/28/22 20:26	87-86-5	
Phenanthrene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 20:26	85-01-8	
Phenol	ND	ug/L	9.6	1.2	1	10/20/22 15:49	10/28/22 20:26	108-95-2	
Pyrene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 20:26	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 20:26	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 20:26	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	36	%	9-74		1	10/20/22 15:49	10/28/22 20:26	367-12-4	
Phenol-d5 (S)	24	%	8-424		1	10/20/22 15:49	10/28/22 20:26	4165-62-2	
Nitrobenzene-d5 (S)	70	%	15-314		1	10/20/22 15:49	10/28/22 20:26	4165-60-0	
2-Fluorobiphenyl (S)	72	%	32-92		1	10/20/22 15:49	10/28/22 20:26	321-60-8	
2,4,6-Tribromophenol (S)	84	%	27-125		1	10/20/22 15:49	10/28/22 20:26	118-79-6	
p-Terphenyl-d14 (S)	75	%	8-146		1	10/20/22 15:49	10/28/22 20:26	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	10	1		10/19/22 17:44	107-02-8	
Acrylonitrile	ND	ug/L	100	2.4	1		10/19/22 17:44	107-13-1	
Benzene	ND	ug/L	5.0	0.82	1		10/19/22 17:44	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.82	1		10/19/22 17:44	75-27-4	
Bromoform	ND	ug/L	5.0	0.73	1		10/19/22 17:44	75-25-2	
Bromomethane	ND	ug/L	5.0	0.44	1		10/19/22 17:44	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.68	1		10/19/22 17:44	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.95	1		10/19/22 17:44	108-90-7	
Chloroethane	ND	ug/L	5.0	0.63	1		10/19/22 17:44	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	2.7	1		10/19/22 17:44	110-75-8	
Chloroform	1.8J	ug/L	4.8	0.83	1		10/19/22 17:44	67-66-3	
Chloromethane	ND	ug/L	5.0	0.44	1		10/19/22 17:44	74-87-3	
Dibromochloromethane	4.1J	ug/L	5.0	0.89	1		10/19/22 17:44	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.84	1		10/19/22 17:44	75-34-3	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 20-CON-10172022**      **Lab ID: 50328663001**      Collected: 10/17/22 10:55      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.85	1		10/19/22 17:44	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.56	1		10/19/22 17:44	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.72	1		10/19/22 17:44	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.79	1		10/19/22 17:44	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.86	1		10/19/22 17:44	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.92	1		10/19/22 17:44	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.95	1		10/19/22 17:44	100-41-4	
Methylene Chloride	ND	ug/L	5.0	0.70	1		10/19/22 17:44	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.92	1		10/19/22 17:44	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.75	1		10/19/22 17:44	127-18-4	
Toluene	ND	ug/L	5.0	0.86	1		10/19/22 17:44	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.74	1		10/19/22 17:44	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.88	1		10/19/22 17:44	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.80	1		10/19/22 17:44	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.52	1		10/19/22 17:44	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	101	%	91-114		1		10/19/22 17:44	1868-53-7	
4-Bromofluorobenzene (S)	102	%	85-120		1		10/19/22 17:44	460-00-4	
Toluene-d8 (S)	99	%	85-117		1		10/19/22 17:44	2037-26-5	

**335.4 Cyanide, Total**

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	<b>0.0082</b>	mg/L	0.0050	0.0018	1	10/20/22 08:00	10/20/22 13:56	57-12-5	
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**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 20-UPS-10172022** Lab ID: 50328663002 Collected: 10/17/22 10:40 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**608.3 PCB**

Analytical Method: EPA 608.3 Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

PCB-1016 (Aroclor 1016)	ND	ug/L	0.097	0.056	1	10/19/22 18:18	10/25/22 16:22	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.097	0.056	1	10/19/22 18:18	10/25/22 16:22	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.097	0.056	1	10/19/22 18:18	10/25/22 16:22	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.097	0.056	1	10/19/22 18:18	10/25/22 16:22	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.097	0.056	1	10/19/22 18:18	10/25/22 16:22	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.097	0.056	1	10/19/22 18:18	10/25/22 16:22	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.097	0.050	1	10/19/22 18:18	10/25/22 16:22	11096-82-5	

**Surrogates**

Tetrachloro-m-xylene (S)	68	%	1-123		1	10/19/22 18:18	10/25/22 16:22	877-09-8	
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**608.3 Pesticides**

Analytical Method: EPA 608.3 Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

Aldrin	ND	ug/L	0.049	0.0087	1	10/19/22 18:18	10/31/22 15:59	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.049	0.011	1	10/19/22 18:18	10/31/22 15:59	319-84-6	
beta-BHC	ND	ug/L	0.049	0.014	1	10/19/22 18:18	10/31/22 15:59	319-85-7	
delta-BHC	ND	ug/L	0.049	0.013	1	10/19/22 18:18	10/31/22 15:59	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.049	0.012	1	10/19/22 18:18	10/31/22 15:59	58-89-9	
Chlordane (Technical)	ND	ug/L	0.49	0.26	1	10/19/22 18:18	10/31/22 15:59	57-74-9	
4,4'-DDD	ND	ug/L	0.097	0.023	1	10/19/22 18:18	10/31/22 15:59	72-54-8	
4,4'-DDE	ND	ug/L	0.097	0.017	1	10/19/22 18:18	10/31/22 15:59	72-55-9	
4,4'-DDT	ND	ug/L	0.097	0.035	1	10/19/22 18:18	10/31/22 15:59	50-29-3	
Dieldrin	ND	ug/L	0.097	0.021	1	10/19/22 18:18	10/31/22 15:59	60-57-1	
Endosulfan I	ND	ug/L	0.049	0.012	1	10/19/22 18:18	10/31/22 15:59	959-98-8	
Endosulfan II	ND	ug/L	0.097	0.024	1	10/19/22 18:18	10/31/22 15:59	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.097	0.020	1	10/19/22 18:18	10/31/22 15:59	1031-07-8	
Endrin	ND	ug/L	0.097	0.026	1	10/19/22 18:18	10/31/22 15:59	72-20-8	
Endrin aldehyde	ND	ug/L	0.097	0.024	1	10/19/22 18:18	10/31/22 15:59	7421-93-4	
Heptachlor	ND	ug/L	0.049	0.0097	1	10/19/22 18:18	10/31/22 15:59	76-44-8	
Heptachlor epoxide	ND	ug/L	0.049	0.011	1	10/19/22 18:18	10/31/22 15:59	1024-57-3	
Toxaphene	ND	ug/L	0.97	0.35	1	10/19/22 18:18	10/31/22 15:59	8001-35-2	

**Surrogates**

Decachlorobiphenyl (S)	93	%	1-140		1	10/19/22 18:18	10/31/22 15:59	2051-24-3	
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**200.8 Metals, Total ICPMS**

Analytical Method: EPA 200.8 Preparation Method: EPA 200.8  
Pace Analytical Services - Indianapolis

Antimony	<b>0.00031J</b>	mg/L	0.0010	0.00013	1	10/19/22 08:00	10/20/22 13:31	7440-36-0	
Arsenic	<b>0.00095J</b>	mg/L	0.0010	0.00011	1	10/19/22 08:00	10/20/22 13:31	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	10/19/22 08:00	10/20/22 13:31	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	10/19/22 08:00	10/20/22 13:31	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	10/19/22 08:00	10/20/22 13:31	7440-47-3	
Copper	<b>0.0019</b>	mg/L	0.0010	0.00037	1	10/19/22 08:00	10/20/22 13:31	7440-50-8	
Lead	<b>0.00019J</b>	mg/L	0.0010	0.000080	1	10/19/22 08:00	10/20/22 13:31	7439-92-1	
Nickel	<b>0.0027</b>	mg/L	0.00050	0.00039	1	10/19/22 08:00	10/20/22 13:31	7440-02-0	
Selenium	<b>0.0011</b>	mg/L	0.0010	0.00035	1	10/19/22 08:00	10/20/22 13:31	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	10/19/22 08:00	10/20/22 13:31	7440-22-4	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

**Sample: 20-UPS-10172022**      **Lab ID: 50328663002**      Collected: 10/17/22 10:40      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	ND	mg/L	0.0010	0.000073	1	10/19/22 08:00	10/20/22 13:31	7440-28-0	
Zinc	<b>0.019</b>	mg/L	0.0030	0.0010	1	10/19/22 08:00	10/20/22 13:31	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/23/22 19:34	10/24/22 11:43	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	10.0	1.8	1	10/20/22 15:49	10/28/22 20:42	83-32-9	
Acenaphthylene	ND	ug/L	10.0	1.9	1	10/20/22 15:49	10/28/22 20:42	208-96-8	
Anthracene	ND	ug/L	10.0	1.9	1	10/20/22 15:49	10/28/22 20:42	120-12-7	
Benzdine	ND	ug/L	50.0	6.0	1	10/20/22 15:49	10/28/22 20:42	92-87-5	
Benzo(a)anthracene	ND	ug/L	10.0	1.7	1	10/20/22 15:49	10/28/22 20:42	56-55-3	
Benzo(a)pyrene	ND	ug/L	10.0	2.3	1	10/20/22 15:49	10/28/22 20:42	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	10.0	2.6	1	10/20/22 15:49	10/28/22 20:42	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	10.0	2.2	1	10/20/22 15:49	10/28/22 20:42	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	10.0	2.6	1	10/20/22 15:49	10/28/22 20:42	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	10.0	2.9	1	10/20/22 15:49	10/28/22 20:42	101-55-3	
Butylbenzylphthalate	ND	ug/L	10.0	3.7	1	10/20/22 15:49	10/28/22 20:42	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	20.0	3.0	1	10/20/22 15:49	10/28/22 20:42	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	10.0	3.1	1	10/20/22 15:49	10/28/22 20:42	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	10.0	2.2	1	10/20/22 15:49	10/28/22 20:42	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	10.0	3.6	1	10/20/22 15:49	10/28/22 20:42	108-60-1	
2-Chloronaphthalene	ND	ug/L	10.0	2.6	1	10/20/22 15:49	10/28/22 20:42	91-58-7	
2-Chlorophenol	ND	ug/L	10.0	2.3	1	10/20/22 15:49	10/28/22 20:42	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	10.0	2.7	1	10/20/22 15:49	10/28/22 20:42	7005-72-3	
Chrysene	ND	ug/L	10.0	1.9	1	10/20/22 15:49	10/28/22 20:42	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	10.0	2.6	1	10/20/22 15:49	10/28/22 20:42	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	10.0	2.7	1	10/20/22 15:49	10/28/22 20:42	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	10.0	2.9	1	10/20/22 15:49	10/28/22 20:42	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	10.0	2.9	1	10/20/22 15:49	10/28/22 20:42	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	20.0	3.1	1	10/20/22 15:49	10/28/22 20:42	91-94-1	
2,4-Dichlorophenol	ND	ug/L	10.0	2.3	1	10/20/22 15:49	10/28/22 20:42	120-83-2	
Diethylphthalate	ND	ug/L	10.0	3.2	1	10/20/22 15:49	10/28/22 20:42	84-66-2	
2,4-Dimethylphenol	ND	ug/L	10.0	2.3	1	10/20/22 15:49	10/28/22 20:42	105-67-9	
Dimethylphthalate	ND	ug/L	10.0	2.3	1	10/20/22 15:49	10/28/22 20:42	131-11-3	
Di-n-butylphthalate	ND	ug/L	10.0	3.3	1	10/20/22 15:49	10/28/22 20:42	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	50.0	8.2	1	10/20/22 15:49	10/28/22 20:42	534-52-1	
2,4-Dinitrophenol	ND	ug/L	50.0	5.2	1	10/20/22 15:49	10/28/22 20:42	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	10.0	2.4	1	10/20/22 15:49	10/28/22 20:42	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	10.0	2.9	1	10/20/22 15:49	10/28/22 20:42	606-20-2	
Di-n-octylphthalate	ND	ug/L	10.0	7.3	1	10/20/22 15:49	10/28/22 20:42	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	10.0	2.1	1	10/20/22 15:49	10/28/22 20:42	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	5.0	4.2	1	10/20/22 15:49	10/28/22 20:42	117-81-7	

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 20-UPS-10172022 Lab ID: 50328663002 Collected: 10/17/22 10:40 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	10.0	1.8	1	10/20/22 15:49	10/28/22 20:42	206-44-0	
Fluorene	ND	ug/L	10.0	2.0	1	10/20/22 15:49	10/28/22 20:42	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	10.0	3.8	1	10/20/22 15:49	10/28/22 20:42	87-68-3	
Hexachlorobenzene	ND	ug/L	10.0	3.3	1	10/20/22 15:49	10/28/22 20:42	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	10.0	4.5	1	10/20/22 15:49	10/28/22 20:42	77-47-4	N2
Hexachloroethane	ND	ug/L	10.0	3.4	1	10/20/22 15:49	10/28/22 20:42	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	10.0	2.6	1	10/20/22 15:49	10/28/22 20:42	193-39-5	
Isophorone	ND	ug/L	10.0	1.9	1	10/20/22 15:49	10/28/22 20:42	78-59-1	
Naphthalene	ND	ug/L	10.0	2.4	1	10/20/22 15:49	10/28/22 20:42	91-20-3	
Nitrobenzene	ND	ug/L	10.0	3.1	1	10/20/22 15:49	10/28/22 20:42	98-95-3	
2-Nitrophenol	ND	ug/L	10.0	4.2	1	10/20/22 15:49	10/28/22 20:42	88-75-5	
4-Nitrophenol	ND	ug/L	50.0	5.1	1	10/20/22 15:49	10/28/22 20:42	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	20.0	3.5	1	10/20/22 15:49	10/28/22 20:42	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	10.0	3.0	1	10/20/22 15:49	10/28/22 20:42	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	10.0	2.2	1	10/20/22 15:49	10/28/22 20:42	86-30-6	
Pentachlorophenol	ND	ug/L	50.0	6.7	1	10/20/22 15:49	10/28/22 20:42	87-86-5	
Phenanthrene	ND	ug/L	10.0	1.9	1	10/20/22 15:49	10/28/22 20:42	85-01-8	
Phenol	ND	ug/L	10.0	1.2	1	10/20/22 15:49	10/28/22 20:42	108-95-2	
Pyrene	ND	ug/L	10.0	2.0	1	10/20/22 15:49	10/28/22 20:42	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	10.0	3.7	1	10/20/22 15:49	10/28/22 20:42	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	10.0	2.8	1	10/20/22 15:49	10/28/22 20:42	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	36	%	9-74		1	10/20/22 15:49	10/28/22 20:42	367-12-4	
Phenol-d5 (S)	24	%	8-424		1	10/20/22 15:49	10/28/22 20:42	4165-62-2	
Nitrobenzene-d5 (S)	69	%	15-314		1	10/20/22 15:49	10/28/22 20:42	4165-60-0	
2-Fluorobiphenyl (S)	70	%	32-92		1	10/20/22 15:49	10/28/22 20:42	321-60-8	
2,4,6-Tribromophenol (S)	86	%	27-125		1	10/20/22 15:49	10/28/22 20:42	118-79-6	
p-Terphenyl-d14 (S)	46	%	8-146		1	10/20/22 15:49	10/28/22 20:42	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	10	1		10/19/22 18:17	107-02-8	
Acrylonitrile	ND	ug/L	100	2.4	1		10/19/22 18:17	107-13-1	
Benzene	ND	ug/L	5.0	0.82	1		10/19/22 18:17	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.82	1		10/19/22 18:17	75-27-4	
Bromoform	ND	ug/L	5.0	0.73	1		10/19/22 18:17	75-25-2	
Bromomethane	ND	ug/L	5.0	0.44	1		10/19/22 18:17	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.68	1		10/19/22 18:17	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.95	1		10/19/22 18:17	108-90-7	
Chloroethane	ND	ug/L	5.0	0.63	1		10/19/22 18:17	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	2.7	1		10/19/22 18:17	110-75-8	
Chloroform	ND	ug/L	4.8	0.83	1		10/19/22 18:17	67-66-3	
Chloromethane	ND	ug/L	5.0	0.44	1		10/19/22 18:17	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.89	1		10/19/22 18:17	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.84	1		10/19/22 18:17	75-34-3	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 20-UPS-10172022**      **Lab ID: 50328663002**      Collected: 10/17/22 10:40      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.85	1		10/19/22 18:17	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.56	1		10/19/22 18:17	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.72	1		10/19/22 18:17	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.79	1		10/19/22 18:17	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.86	1		10/19/22 18:17	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.92	1		10/19/22 18:17	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.95	1		10/19/22 18:17	100-41-4	
Methylene Chloride	ND	ug/L	5.0	0.70	1		10/19/22 18:17	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.92	1		10/19/22 18:17	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.75	1		10/19/22 18:17	127-18-4	
Toluene	ND	ug/L	5.0	0.86	1		10/19/22 18:17	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.74	1		10/19/22 18:17	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.88	1		10/19/22 18:17	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.80	1		10/19/22 18:17	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.52	1		10/19/22 18:17	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	102	%	91-114		1		10/19/22 18:17	1868-53-7	
4-Bromofluorobenzene (S)	102	%	85-120		1		10/19/22 18:17	460-00-4	
Toluene-d8 (S)	99	%	85-117		1		10/19/22 18:17	2037-26-5	

**335.4 Cyanide, Total**

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	ND	mg/L	0.0050	0.0018	1	10/20/22 08:00	10/20/22 13:58	57-12-5	
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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 20-EFF-10172022**      **Lab ID: 50328663003**      Collected: 10/17/22 10:20      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**608.3 PCB**      Analytical Method: EPA 608.3      Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

PCB-1016 (Aroclor 1016)	ND	ug/L	0.10	0.058	1	10/19/22 18:18	10/25/22 16:37	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.10	0.058	1	10/19/22 18:18	10/25/22 16:37	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.10	0.058	1	10/19/22 18:18	10/25/22 16:37	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.10	0.058	1	10/19/22 18:18	10/25/22 16:37	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.10	0.058	1	10/19/22 18:18	10/25/22 16:37	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.10	0.058	1	10/19/22 18:18	10/25/22 16:37	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.10	0.051	1	10/19/22 18:18	10/25/22 16:37	11096-82-5	

**Surrogates**

Tetrachloro-m-xylene (S)	66	%	1-123		1	10/19/22 18:18	10/25/22 16:37	877-09-8	
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**608.3 Pesticides**      Analytical Method: EPA 608.3      Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

Aldrin	ND	ug/L	0.050	0.0090	1	10/19/22 18:18	10/31/22 16:12	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.050	0.011	1	10/19/22 18:18	10/31/22 16:12	319-84-6	
beta-BHC	ND	ug/L	0.050	0.014	1	10/19/22 18:18	10/31/22 16:12	319-85-7	
delta-BHC	ND	ug/L	0.050	0.013	1	10/19/22 18:18	10/31/22 16:12	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.050	0.012	1	10/19/22 18:18	10/31/22 16:12	58-89-9	
Chlordane (Technical)	ND	ug/L	0.50	0.27	1	10/19/22 18:18	10/31/22 16:12	57-74-9	
4,4'-DDD	ND	ug/L	0.10	0.024	1	10/19/22 18:18	10/31/22 16:12	72-54-8	
4,4'-DDE	ND	ug/L	0.10	0.018	1	10/19/22 18:18	10/31/22 16:12	72-55-9	
4,4'-DDT	ND	ug/L	0.10	0.036	1	10/19/22 18:18	10/31/22 16:12	50-29-3	
Dieldrin	ND	ug/L	0.10	0.022	1	10/19/22 18:18	10/31/22 16:12	60-57-1	
Endosulfan I	ND	ug/L	0.050	0.012	1	10/19/22 18:18	10/31/22 16:12	959-98-8	
Endosulfan II	ND	ug/L	0.10	0.025	1	10/19/22 18:18	10/31/22 16:12	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.10	0.021	1	10/19/22 18:18	10/31/22 16:12	1031-07-8	
Endrin	ND	ug/L	0.10	0.027	1	10/19/22 18:18	10/31/22 16:12	72-20-8	
Endrin aldehyde	ND	ug/L	0.10	0.025	1	10/19/22 18:18	10/31/22 16:12	7421-93-4	
Heptachlor	ND	ug/L	0.050	0.010	1	10/19/22 18:18	10/31/22 16:12	76-44-8	
Heptachlor epoxide	ND	ug/L	0.050	0.011	1	10/19/22 18:18	10/31/22 16:12	1024-57-3	
Toxaphene	ND	ug/L	1.0	0.36	1	10/19/22 18:18	10/31/22 16:12	8001-35-2	

**Surrogates**

Decachlorobiphenyl (S)	58	%	1-140		1	10/19/22 18:18	10/31/22 16:12	2051-24-3	
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**200.8 Metals, Total ICMS**      Analytical Method: EPA 200.8      Preparation Method: EPA 200.8  
Pace Analytical Services - Indianapolis

Antimony	<b>0.0010J</b>	mg/L	0.0010	0.00013	1	10/19/22 08:00	10/20/22 13:35	7440-36-0	
Arsenic	<b>0.0096</b>	mg/L	0.0010	0.00011	1	10/19/22 08:00	10/20/22 13:35	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	10/19/22 08:00	10/20/22 13:35	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	10/19/22 08:00	10/20/22 13:35	7440-43-9	
Chromium	<b>0.00066J</b>	mg/L	0.0020	0.00063	1	10/19/22 08:00	10/20/22 13:35	7440-47-3	
Copper	<b>0.0069</b>	mg/L	0.0010	0.00037	1	10/19/22 08:00	10/20/22 13:35	7440-50-8	
Lead	ND	mg/L	0.0010	0.000080	1	10/19/22 08:00	10/20/22 13:35	7439-92-1	
Nickel	<b>0.0045</b>	mg/L	0.00050	0.00039	1	10/19/22 08:00	10/20/22 13:35	7440-02-0	
Selenium	<b>0.091</b>	mg/L	0.0010	0.00035	1	10/19/22 08:00	10/20/22 13:35	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	10/19/22 08:00	10/20/22 13:35	7440-22-4	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 20-EFF-10172022**      **Lab ID: 50328663003**      Collected: 10/17/22 10:20      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	0.00025J	mg/L	0.0010	0.000073	1	10/19/22 08:00	10/20/22 13:35	7440-28-0	
Zinc	0.013	mg/L	0.0030	0.0010	1	10/19/22 08:00	10/20/22 13:35	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/23/22 19:34	10/24/22 11:46	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.7	1.7	1	10/20/22 15:49	10/28/22 20:59	83-32-9	
Acenaphthylene	ND	ug/L	9.7	1.8	1	10/20/22 15:49	10/28/22 20:59	208-96-8	
Anthracene	ND	ug/L	9.7	1.8	1	10/20/22 15:49	10/28/22 20:59	120-12-7	
Benzdine	ND	ug/L	48.5	5.8	1	10/20/22 15:49	10/28/22 20:59	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.7	1.6	1	10/20/22 15:49	10/28/22 20:59	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.7	2.2	1	10/20/22 15:49	10/28/22 20:59	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.7	2.5	1	10/20/22 15:49	10/28/22 20:59	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.7	2.1	1	10/20/22 15:49	10/28/22 20:59	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.7	2.5	1	10/20/22 15:49	10/28/22 20:59	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.7	2.8	1	10/20/22 15:49	10/28/22 20:59	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.7	3.6	1	10/20/22 15:49	10/28/22 20:59	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.4	2.9	1	10/20/22 15:49	10/28/22 20:59	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.7	3.0	1	10/20/22 15:49	10/28/22 20:59	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.7	2.1	1	10/20/22 15:49	10/28/22 20:59	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.7	3.5	1	10/20/22 15:49	10/28/22 20:59	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.7	2.5	1	10/20/22 15:49	10/28/22 20:59	91-58-7	
2-Chlorophenol	ND	ug/L	9.7	2.2	1	10/20/22 15:49	10/28/22 20:59	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.7	2.6	1	10/20/22 15:49	10/28/22 20:59	7005-72-3	
Chrysene	ND	ug/L	9.7	1.8	1	10/20/22 15:49	10/28/22 20:59	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.7	2.5	1	10/20/22 15:49	10/28/22 20:59	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.7	2.6	1	10/20/22 15:49	10/28/22 20:59	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.7	2.8	1	10/20/22 15:49	10/28/22 20:59	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.7	2.9	1	10/20/22 15:49	10/28/22 20:59	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.4	3.1	1	10/20/22 15:49	10/28/22 20:59	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.7	2.2	1	10/20/22 15:49	10/28/22 20:59	120-83-2	
Diethylphthalate	ND	ug/L	9.7	3.1	1	10/20/22 15:49	10/28/22 20:59	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.7	2.2	1	10/20/22 15:49	10/28/22 20:59	105-67-9	
Dimethylphthalate	ND	ug/L	9.7	2.3	1	10/20/22 15:49	10/28/22 20:59	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.7	3.2	1	10/20/22 15:49	10/28/22 20:59	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	48.5	7.9	1	10/20/22 15:49	10/28/22 20:59	534-52-1	
2,4-Dinitrophenol	ND	ug/L	48.5	5.0	1	10/20/22 15:49	10/28/22 20:59	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.7	2.3	1	10/20/22 15:49	10/28/22 20:59	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.7	2.8	1	10/20/22 15:49	10/28/22 20:59	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.7	7.1	1	10/20/22 15:49	10/28/22 20:59	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.7	2.0	1	10/20/22 15:49	10/28/22 20:59	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.9	4.1	1	10/20/22 15:49	10/28/22 20:59	117-81-7	

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 20-EFF-10172022 Lab ID: 50328663003 Collected: 10/17/22 10:20 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	9.7	1.8	1	10/20/22 15:49	10/28/22 20:59	206-44-0	
Fluorene	ND	ug/L	9.7	1.9	1	10/20/22 15:49	10/28/22 20:59	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.7	3.7	1	10/20/22 15:49	10/28/22 20:59	87-68-3	
Hexachlorobenzene	ND	ug/L	9.7	3.2	1	10/20/22 15:49	10/28/22 20:59	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.4	4.4	1	10/20/22 15:49	10/28/22 20:59	77-47-4	N2
Hexachloroethane	ND	ug/L	9.7	3.3	1	10/20/22 15:49	10/28/22 20:59	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.7	2.5	1	10/20/22 15:49	10/28/22 20:59	193-39-5	
Isophorone	ND	ug/L	9.7	1.9	1	10/20/22 15:49	10/28/22 20:59	78-59-1	
Naphthalene	ND	ug/L	9.7	2.3	1	10/20/22 15:49	10/28/22 20:59	91-20-3	
Nitrobenzene	ND	ug/L	9.7	3.0	1	10/20/22 15:49	10/28/22 20:59	98-95-3	
2-Nitrophenol	ND	ug/L	9.7	4.1	1	10/20/22 15:49	10/28/22 20:59	88-75-5	
4-Nitrophenol	ND	ug/L	48.5	5.0	1	10/20/22 15:49	10/28/22 20:59	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.4	3.4	1	10/20/22 15:49	10/28/22 20:59	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.7	2.9	1	10/20/22 15:49	10/28/22 20:59	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.7	2.1	1	10/20/22 15:49	10/28/22 20:59	86-30-6	
Pentachlorophenol	ND	ug/L	48.5	6.5	1	10/20/22 15:49	10/28/22 20:59	87-86-5	
Phenanthrene	ND	ug/L	9.7	1.9	1	10/20/22 15:49	10/28/22 20:59	85-01-8	
Phenol	ND	ug/L	9.7	1.2	1	10/20/22 15:49	10/28/22 20:59	108-95-2	
Pyrene	ND	ug/L	9.7	2.0	1	10/20/22 15:49	10/28/22 20:59	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.7	3.6	1	10/20/22 15:49	10/28/22 20:59	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.7	2.7	1	10/20/22 15:49	10/28/22 20:59	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	26	%	9-74		1	10/20/22 15:49	10/28/22 20:59	367-12-4	
Phenol-d5 (S)	19	%	8-424		1	10/20/22 15:49	10/28/22 20:59	4165-62-2	
Nitrobenzene-d5 (S)	53	%	15-314		1	10/20/22 15:49	10/28/22 20:59	4165-60-0	
2-Fluorobiphenyl (S)	59	%	32-92		1	10/20/22 15:49	10/28/22 20:59	321-60-8	
2,4,6-Tribromophenol (S)	76	%	27-125		1	10/20/22 15:49	10/28/22 20:59	118-79-6	
p-Terphenyl-d14 (S)	64	%	8-146		1	10/20/22 15:49	10/28/22 20:59	1718-51-0	
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
Acrolein	ND	ug/L	50.0	10	1		10/19/22 18:49	107-02-8	
Acrylonitrile	ND	ug/L	100	2.4	1		10/19/22 18:49	107-13-1	
Benzene	ND	ug/L	5.0	0.82	1		10/19/22 18:49	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.82	1		10/19/22 18:49	75-27-4	
Bromoform	ND	ug/L	5.0	0.73	1		10/19/22 18:49	75-25-2	
Bromomethane	ND	ug/L	5.0	0.44	1		10/19/22 18:49	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.68	1		10/19/22 18:49	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.95	1		10/19/22 18:49	108-90-7	
Chloroethane	ND	ug/L	5.0	0.63	1		10/19/22 18:49	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	2.7	1		10/19/22 18:49	110-75-8	
Chloroform	ND	ug/L	4.8	0.83	1		10/19/22 18:49	67-66-3	
Chloromethane	ND	ug/L	5.0	0.44	1		10/19/22 18:49	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.89	1		10/19/22 18:49	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.84	1		10/19/22 18:49	75-34-3	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 20-EFF-10172022**      **Lab ID: 50328663003**      Collected: 10/17/22 10:20      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.85	1		10/19/22 18:49	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.56	1		10/19/22 18:49	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.72	1		10/19/22 18:49	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.79	1		10/19/22 18:49	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.86	1		10/19/22 18:49	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.92	1		10/19/22 18:49	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.95	1		10/19/22 18:49	100-41-4	
Methylene Chloride	ND	ug/L	5.0	0.70	1		10/19/22 18:49	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.92	1		10/19/22 18:49	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.75	1		10/19/22 18:49	127-18-4	
Toluene	ND	ug/L	5.0	0.86	1		10/19/22 18:49	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.74	1		10/19/22 18:49	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.88	1		10/19/22 18:49	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.80	1		10/19/22 18:49	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.52	1		10/19/22 18:49	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	102	%	91-114		1		10/19/22 18:49	1868-53-7	
4-Bromofluorobenzene (S)	104	%	85-120		1		10/19/22 18:49	460-00-4	
Toluene-d8 (S)	97	%	85-117		1		10/19/22 18:49	2037-26-5	
<b>335.4 Cyanide, Total</b>									
Analytical Method: EPA 335.4      Preparation Method: EPA 335.4									
Pace Analytical Services - Indianapolis									
Cyanide	<b>0.0044J</b>	mg/L	0.0050	0.0018	1	10/20/22 08:00	10/20/22 13:58	57-12-5	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 20-DNS-10172022 Lab ID: 50328663004 Collected: 10/17/22 09:50 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**608.3 PCB**

Analytical Method: EPA 608.3 Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

PCB-1016 (Aroclor 1016)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:52	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:52	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:52	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:52	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:52	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 16:52	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.096	0.049	1	10/19/22 18:18	10/25/22 16:52	11096-82-5	

**Surrogates**

Tetrachloro-m-xylene (S)	67	%	1-123		1	10/19/22 18:18	10/25/22 16:52	877-09-8	
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**608.3 Pesticides**

Analytical Method: EPA 608.3 Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

Aldrin	ND	ug/L	0.048	0.0087	1	10/19/22 18:18	10/31/22 16:25	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 16:25	319-84-6	
beta-BHC	ND	ug/L	0.048	0.013	1	10/19/22 18:18	10/31/22 16:25	319-85-7	
delta-BHC	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 16:25	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 16:25	58-89-9	
Chlordane (Technical)	ND	ug/L	0.48	0.26	1	10/19/22 18:18	10/31/22 16:25	57-74-9	
4,4'-DDD	ND	ug/L	0.096	0.023	1	10/19/22 18:18	10/31/22 16:25	72-54-8	
4,4'-DDE	ND	ug/L	0.096	0.017	1	10/19/22 18:18	10/31/22 16:25	72-55-9	
4,4'-DDT	ND	ug/L	0.096	0.035	1	10/19/22 18:18	10/31/22 16:25	50-29-3	
Dieldrin	ND	ug/L	0.096	0.021	1	10/19/22 18:18	10/31/22 16:25	60-57-1	
Endosulfan I	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 16:25	959-98-8	
Endosulfan II	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 16:25	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.096	0.020	1	10/19/22 18:18	10/31/22 16:25	1031-07-8	
Endrin	ND	ug/L	0.096	0.026	1	10/19/22 18:18	10/31/22 16:25	72-20-8	
Endrin aldehyde	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 16:25	7421-93-4	
Heptachlor	ND	ug/L	0.048	0.0096	1	10/19/22 18:18	10/31/22 16:25	76-44-8	
Heptachlor epoxide	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 16:25	1024-57-3	
Toxaphene	ND	ug/L	0.96	0.35	1	10/19/22 18:18	10/31/22 16:25	8001-35-2	

**Surrogates**

Decachlorobiphenyl (S)	36	%	1-140		1	10/19/22 18:18	10/31/22 16:25	2051-24-3	
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**200.8 Metals, Total ICPMS**

Analytical Method: EPA 200.8 Preparation Method: EPA 200.8  
Pace Analytical Services - Indianapolis

Antimony	<b>0.00074J</b>	mg/L	0.0010	0.00013	1	10/19/22 08:00	10/20/22 13:39	7440-36-0	
Arsenic	<b>0.0047</b>	mg/L	0.0010	0.00011	1	10/19/22 08:00	10/20/22 13:39	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	10/19/22 08:00	10/20/22 13:39	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	10/19/22 08:00	10/20/22 13:39	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	10/19/22 08:00	10/20/22 13:39	7440-47-3	
Copper	<b>0.0049</b>	mg/L	0.0010	0.00037	1	10/19/22 08:00	10/20/22 13:39	7440-50-8	
Lead	<b>0.00016J</b>	mg/L	0.0010	0.000080	1	10/19/22 08:00	10/20/22 13:39	7439-92-1	
Nickel	<b>0.0035</b>	mg/L	0.00050	0.00039	1	10/19/22 08:00	10/20/22 13:39	7440-02-0	
Selenium	<b>0.059</b>	mg/L	0.0010	0.00035	1	10/19/22 08:00	10/20/22 13:39	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	10/19/22 08:00	10/20/22 13:39	7440-22-4	

**REPORT OF LABORATORY ANALYSIS**

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### ANALYTICAL RESULTS

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 20-DNS-10172022 Lab ID: 50328663004 Collected: 10/17/22 09:50 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	0.00012J	mg/L	0.0010	0.000073	1	10/19/22 08:00	10/20/22 13:39	7440-28-0	
Zinc	0.0070	mg/L	0.0030	0.0010	1	10/19/22 08:00	10/20/22 13:39	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/23/22 19:34	10/24/22 11:48	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.6	1.7	1	10/20/22 15:49	10/28/22 21:15	83-32-9	
Acenaphthylene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:15	208-96-8	
Anthracene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:15	120-12-7	
Benzdine	ND	ug/L	48.1	5.8	1	10/20/22 15:49	10/28/22 21:15	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.6	1.6	1	10/20/22 15:49	10/28/22 21:15	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:15	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:15	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 21:15	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:15	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 21:15	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.6	3.6	1	10/20/22 15:49	10/28/22 21:15	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.2	2.9	1	10/20/22 15:49	10/28/22 21:15	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 21:15	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 21:15	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 21:15	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:15	91-58-7	
2-Chlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:15	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 21:15	7005-72-3	
Chrysene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:15	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:15	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 21:15	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 21:15	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 21:15	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.2	3.0	1	10/20/22 15:49	10/28/22 21:15	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:15	120-83-2	
Diethylphthalate	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 21:15	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:15	105-67-9	
Dimethylphthalate	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:15	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 21:15	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	48.1	7.8	1	10/20/22 15:49	10/28/22 21:15	534-52-1	
2,4-Dinitrophenol	ND	ug/L	48.1	5.0	1	10/20/22 15:49	10/28/22 21:15	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 21:15	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 21:15	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.6	7.1	1	10/20/22 15:49	10/28/22 21:15	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.6	2.0	1	10/20/22 15:49	10/28/22 21:15	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.8	4.0	1	10/20/22 15:49	10/28/22 21:15	117-81-7	

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 20-DNS-10172022 Lab ID: 50328663004 Collected: 10/17/22 09:50 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**625.1 MSSV**

Analytical Method: EPA 625.1 Preparation Method: EPA 625.1  
Pace Analytical Services - Indianapolis

Fluoranthene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:15	206-44-0	
Fluorene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 21:15	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.6	3.7	1	10/20/22 15:49	10/28/22 21:15	87-68-3	
Hexachlorobenzene	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 21:15	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.2	4.4	1	10/20/22 15:49	10/28/22 21:15	77-47-4	N2
Hexachloroethane	ND	ug/L	9.6	3.3	1	10/20/22 15:49	10/28/22 21:15	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:15	193-39-5	
Isophorone	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 21:15	78-59-1	
Naphthalene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 21:15	91-20-3	
Nitrobenzene	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 21:15	98-95-3	
2-Nitrophenol	ND	ug/L	9.6	4.0	1	10/20/22 15:49	10/28/22 21:15	88-75-5	
4-Nitrophenol	ND	ug/L	48.1	4.9	1	10/20/22 15:49	10/28/22 21:15	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.2	3.4	1	10/20/22 15:49	10/28/22 21:15	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 21:15	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 21:15	86-30-6	
Pentachlorophenol	ND	ug/L	48.1	6.5	1	10/20/22 15:49	10/28/22 21:15	87-86-5	
Phenanthrene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:15	85-01-8	
Phenol	ND	ug/L	9.6	1.2	1	10/20/22 15:49	10/28/22 21:15	108-95-2	
Pyrene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 21:15	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 21:15	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 21:15	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	27	%	9-74		1	10/20/22 15:49	10/28/22 21:15	367-12-4	
Phenol-d5 (S)	18	%	8-424		1	10/20/22 15:49	10/28/22 21:15	4165-62-2	
Nitrobenzene-d5 (S)	65	%	15-314		1	10/20/22 15:49	10/28/22 21:15	4165-60-0	
2-Fluorobiphenyl (S)	69	%	32-92		1	10/20/22 15:49	10/28/22 21:15	321-60-8	
2,4,6-Tribromophenol (S)	84	%	27-125		1	10/20/22 15:49	10/28/22 21:15	118-79-6	
p-Terphenyl-d14 (S)	65	%	8-146		1	10/20/22 15:49	10/28/22 21:15	1718-51-0	

**624.1 Volatile Organics**

Analytical Method: EPA 624.1  
Pace Analytical Services - Indianapolis

Acrolein	ND	ug/L	50.0	10	1		10/19/22 19:22	107-02-8	
Acrylonitrile	ND	ug/L	100	2.4	1		10/19/22 19:22	107-13-1	
Benzene	ND	ug/L	5.0	0.82	1		10/19/22 19:22	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.82	1		10/19/22 19:22	75-27-4	
Bromoform	ND	ug/L	5.0	0.73	1		10/19/22 19:22	75-25-2	
Bromomethane	ND	ug/L	5.0	0.44	1		10/19/22 19:22	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.68	1		10/19/22 19:22	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.95	1		10/19/22 19:22	108-90-7	
Chloroethane	ND	ug/L	5.0	0.63	1		10/19/22 19:22	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	2.7	1		10/19/22 19:22	110-75-8	
Chloroform	ND	ug/L	4.8	0.83	1		10/19/22 19:22	67-66-3	
Chloromethane	ND	ug/L	5.0	0.44	1		10/19/22 19:22	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.89	1		10/19/22 19:22	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.84	1		10/19/22 19:22	75-34-3	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 20-DNS-10172022**      **Lab ID: 50328663004**      Collected: 10/17/22 09:50      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.85	1		10/19/22 19:22	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.56	1		10/19/22 19:22	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.72	1		10/19/22 19:22	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.79	1		10/19/22 19:22	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.86	1		10/19/22 19:22	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.92	1		10/19/22 19:22	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.95	1		10/19/22 19:22	100-41-4	
Methylene Chloride	ND	ug/L	5.0	0.70	1		10/19/22 19:22	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.92	1		10/19/22 19:22	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.75	1		10/19/22 19:22	127-18-4	
Toluene	ND	ug/L	5.0	0.86	1		10/19/22 19:22	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.74	1		10/19/22 19:22	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.88	1		10/19/22 19:22	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.80	1		10/19/22 19:22	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.52	1		10/19/22 19:22	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	103	%	91-114		1		10/19/22 19:22	1868-53-7	
4-Bromofluorobenzene (S)	104	%	85-120		1		10/19/22 19:22	460-00-4	
Toluene-d8 (S)	100	%	85-117		1		10/19/22 19:22	2037-26-5	
<b>335.4 Cyanide, Total</b>									
Analytical Method: EPA 335.4      Preparation Method: EPA 335.4									
Pace Analytical Services - Indianapolis									
Cyanide	<b>0.0028J</b>	mg/L	0.0050	0.0018	1	10/20/22 08:00	10/20/22 14:03	57-12-5	

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 30-CON-10172022**      **Lab ID: 50328663005**      Collected: 10/17/22 12:45      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**608.3 PCB**      Analytical Method: EPA 608.3      Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

PCB-1016 (Aroclor 1016)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:06	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:06	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:06	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:06	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:06	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:06	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.096	0.049	1	10/19/22 18:18	10/25/22 17:06	11096-82-5	

**Surrogates**

Tetrachloro-m-xylene (S)	68	%	1-123		1	10/19/22 18:18	10/25/22 17:06	877-09-8	
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**608.3 Pesticides**      Analytical Method: EPA 608.3      Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

Aldrin	ND	ug/L	0.048	0.0087	1	10/19/22 18:18	10/31/22 16:37	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 16:37	319-84-6	
beta-BHC	ND	ug/L	0.048	0.013	1	10/19/22 18:18	10/31/22 16:37	319-85-7	
delta-BHC	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 16:37	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 16:37	58-89-9	
Chlordane (Technical)	ND	ug/L	0.48	0.26	1	10/19/22 18:18	10/31/22 16:37	57-74-9	
4,4'-DDD	ND	ug/L	0.096	0.023	1	10/19/22 18:18	10/31/22 16:37	72-54-8	
4,4'-DDE	ND	ug/L	0.096	0.017	1	10/19/22 18:18	10/31/22 16:37	72-55-9	
4,4'-DDT	ND	ug/L	0.096	0.035	1	10/19/22 18:18	10/31/22 16:37	50-29-3	
Dieldrin	ND	ug/L	0.096	0.021	1	10/19/22 18:18	10/31/22 16:37	60-57-1	
Endosulfan I	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 16:37	959-98-8	
Endosulfan II	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 16:37	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.096	0.020	1	10/19/22 18:18	10/31/22 16:37	1031-07-8	
Endrin	ND	ug/L	0.096	0.026	1	10/19/22 18:18	10/31/22 16:37	72-20-8	
Endrin aldehyde	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 16:37	7421-93-4	
Heptachlor	ND	ug/L	0.048	0.0096	1	10/19/22 18:18	10/31/22 16:37	76-44-8	
Heptachlor epoxide	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 16:37	1024-57-3	
Toxaphene	ND	ug/L	0.96	0.35	1	10/19/22 18:18	10/31/22 16:37	8001-35-2	

**Surrogates**

Decachlorobiphenyl (S)	62	%	1-140		1	10/19/22 18:18	10/31/22 16:37	2051-24-3	
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**200.8 Metals, Total ICMS**      Analytical Method: EPA 200.8      Preparation Method: EPA 200.8  
Pace Analytical Services - Indianapolis

Antimony	ND	mg/L	0.0010	0.00013	1	10/19/22 08:00	10/20/22 13:43	7440-36-0	
Arsenic	<b>0.00046J</b>	mg/L	0.0010	0.00011	1	10/19/22 08:00	10/20/22 13:43	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	10/19/22 08:00	10/20/22 13:43	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	10/19/22 08:00	10/20/22 13:43	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	10/19/22 08:00	10/20/22 13:43	7440-47-3	
Copper	<b>0.0040</b>	mg/L	0.0010	0.00037	1	10/19/22 08:00	10/20/22 13:43	7440-50-8	
Lead	ND	mg/L	0.0010	0.000080	1	10/19/22 08:00	10/20/22 13:43	7439-92-1	
Nickel	<b>0.0019</b>	mg/L	0.00050	0.00039	1	10/19/22 08:00	10/20/22 13:43	7440-02-0	
Selenium	<b>0.0029</b>	mg/L	0.0010	0.00035	1	10/19/22 08:00	10/20/22 13:43	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	10/19/22 08:00	10/20/22 13:43	7440-22-4	

**REPORT OF LABORATORY ANALYSIS**

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### ANALYTICAL RESULTS

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 30-CON-10172022 Lab ID: 50328663005 Collected: 10/17/22 12:45 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	ND	mg/L	0.0010	0.000073	1	10/19/22 08:00	10/20/22 13:43	7440-28-0	
Zinc	0.039	mg/L	0.0030	0.0010	1	10/19/22 08:00	10/20/22 13:43	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/23/22 19:34	10/24/22 11:51	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.6	1.7	1	10/20/22 15:49	10/28/22 21:32	83-32-9	
Acenaphthylene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:32	208-96-8	
Anthracene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:32	120-12-7	
Benzdine	ND	ug/L	48.1	5.8	1	10/20/22 15:49	10/28/22 21:32	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.6	1.6	1	10/20/22 15:49	10/28/22 21:32	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:32	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:32	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 21:32	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:32	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 21:32	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.6	3.6	1	10/20/22 15:49	10/28/22 21:32	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.2	2.9	1	10/20/22 15:49	10/28/22 21:32	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 21:32	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 21:32	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 21:32	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:32	91-58-7	
2-Chlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:32	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 21:32	7005-72-3	
Chrysene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:32	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:32	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 21:32	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 21:32	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 21:32	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.2	3.0	1	10/20/22 15:49	10/28/22 21:32	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:32	120-83-2	
Diethylphthalate	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 21:32	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:32	105-67-9	
Dimethylphthalate	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:32	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 21:32	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	48.1	7.8	1	10/20/22 15:49	10/28/22 21:32	534-52-1	
2,4-Dinitrophenol	ND	ug/L	48.1	5.0	1	10/20/22 15:49	10/28/22 21:32	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 21:32	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 21:32	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.6	7.1	1	10/20/22 15:49	10/28/22 21:32	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.6	2.0	1	10/20/22 15:49	10/28/22 21:32	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.8	4.0	1	10/20/22 15:49	10/28/22 21:32	117-81-7	

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 30-CON-10172022 Lab ID: 50328663005 Collected: 10/17/22 12:45 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:32	206-44-0	
Fluorene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 21:32	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.6	3.7	1	10/20/22 15:49	10/28/22 21:32	87-68-3	
Hexachlorobenzene	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 21:32	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.2	4.4	1	10/20/22 15:49	10/28/22 21:32	77-47-4	N2
Hexachloroethane	ND	ug/L	9.6	3.3	1	10/20/22 15:49	10/28/22 21:32	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:32	193-39-5	
Isophorone	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 21:32	78-59-1	
Naphthalene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 21:32	91-20-3	
Nitrobenzene	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 21:32	98-95-3	
2-Nitrophenol	ND	ug/L	9.6	4.0	1	10/20/22 15:49	10/28/22 21:32	88-75-5	
4-Nitrophenol	ND	ug/L	48.1	4.9	1	10/20/22 15:49	10/28/22 21:32	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.2	3.4	1	10/20/22 15:49	10/28/22 21:32	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 21:32	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 21:32	86-30-6	
Pentachlorophenol	ND	ug/L	48.1	6.5	1	10/20/22 15:49	10/28/22 21:32	87-86-5	
Phenanthrene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:32	85-01-8	
Phenol	ND	ug/L	9.6	1.2	1	10/20/22 15:49	10/28/22 21:32	108-95-2	
Pyrene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 21:32	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 21:32	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 21:32	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	22	%	9-74		1	10/20/22 15:49	10/28/22 21:32	367-12-4	
Phenol-d5 (S)	17	%	8-424		1	10/20/22 15:49	10/28/22 21:32	4165-62-2	
Nitrobenzene-d5 (S)	47	%	15-314		1	10/20/22 15:49	10/28/22 21:32	4165-60-0	
2-Fluorobiphenyl (S)	52	%	32-92		1	10/20/22 15:49	10/28/22 21:32	321-60-8	
2,4,6-Tribromophenol (S)	75	%	27-125		1	10/20/22 15:49	10/28/22 21:32	118-79-6	
p-Terphenyl-d14 (S)	71	%	8-146		1	10/20/22 15:49	10/28/22 21:32	1718-51-0	

**624.1 Volatile Organics**

Analytical Method: EPA 624.1

Pace Analytical Services - Indianapolis

Acrolein	ND	ug/L	50.0	10	1		10/19/22 19:54	107-02-8	
Acrylonitrile	ND	ug/L	100	2.4	1		10/19/22 19:54	107-13-1	
Benzene	ND	ug/L	5.0	0.82	1		10/19/22 19:54	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.82	1		10/19/22 19:54	75-27-4	
Bromoform	ND	ug/L	5.0	0.73	1		10/19/22 19:54	75-25-2	
Bromomethane	ND	ug/L	5.0	0.44	1		10/19/22 19:54	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.68	1		10/19/22 19:54	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.95	1		10/19/22 19:54	108-90-7	
Chloroethane	ND	ug/L	5.0	0.63	1		10/19/22 19:54	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	2.7	1		10/19/22 19:54	110-75-8	
Chloroform	ND	ug/L	4.8	0.83	1		10/19/22 19:54	67-66-3	
Chloromethane	ND	ug/L	5.0	0.44	1		10/19/22 19:54	74-87-3	
Dibromochloromethane	1.4J	ug/L	5.0	0.89	1		10/19/22 19:54	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.84	1		10/19/22 19:54	75-34-3	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 30-CON-10172022**      **Lab ID: 50328663005**      Collected: 10/17/22 12:45      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.85	1		10/19/22 19:54	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.56	1		10/19/22 19:54	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.72	1		10/19/22 19:54	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.79	1		10/19/22 19:54	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.86	1		10/19/22 19:54	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.92	1		10/19/22 19:54	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.95	1		10/19/22 19:54	100-41-4	
Methylene Chloride	ND	ug/L	5.0	0.70	1		10/19/22 19:54	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.92	1		10/19/22 19:54	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.75	1		10/19/22 19:54	127-18-4	
Toluene	ND	ug/L	5.0	0.86	1		10/19/22 19:54	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.74	1		10/19/22 19:54	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.88	1		10/19/22 19:54	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.80	1		10/19/22 19:54	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.52	1		10/19/22 19:54	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	103	%	91-114		1		10/19/22 19:54	1868-53-7	
4-Bromofluorobenzene (S)	102	%	85-120		1		10/19/22 19:54	460-00-4	
Toluene-d8 (S)	99	%	85-117		1		10/19/22 19:54	2037-26-5	
<b>335.4 Cyanide, Total</b>									
Analytical Method: EPA 335.4      Preparation Method: EPA 335.4									
Pace Analytical Services - Indianapolis									
Cyanide	<b>0.0024J</b>	mg/L	0.0050	0.0018	1	10/20/22 08:00	10/20/22 14:03	57-12-5	

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 30-UPS-10172022 Lab ID: 50328663006 Collected: 10/17/22 12:25 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>608.3 PCB</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
PCB-1016 (Aroclor 1016)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:21	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:21	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:21	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:21	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:21	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:21	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.096	0.049	1	10/19/22 18:18	10/25/22 17:21	11096-82-5	
<b>Surrogates</b>									
Tetrachloro-m-xylene (S)	61	%	1-123		1	10/19/22 18:18	10/25/22 17:21	877-09-8	
<b>608.3 Pesticides</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
Aldrin	ND	ug/L	0.048	0.0087	1	10/19/22 18:18	10/31/22 16:50	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 16:50	319-84-6	
beta-BHC	ND	ug/L	0.048	0.013	1	10/19/22 18:18	10/31/22 16:50	319-85-7	
delta-BHC	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 16:50	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 16:50	58-89-9	
Chlordane (Technical)	ND	ug/L	0.48	0.26	1	10/19/22 18:18	10/31/22 16:50	57-74-9	
4,4'-DDD	ND	ug/L	0.096	0.023	1	10/19/22 18:18	10/31/22 16:50	72-54-8	
4,4'-DDE	ND	ug/L	0.096	0.017	1	10/19/22 18:18	10/31/22 16:50	72-55-9	
4,4'-DDT	ND	ug/L	0.096	0.035	1	10/19/22 18:18	10/31/22 16:50	50-29-3	
Dieldrin	ND	ug/L	0.096	0.021	1	10/19/22 18:18	10/31/22 16:50	60-57-1	
Endosulfan I	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 16:50	959-98-8	
Endosulfan II	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 16:50	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.096	0.020	1	10/19/22 18:18	10/31/22 16:50	1031-07-8	
Endrin	ND	ug/L	0.096	0.026	1	10/19/22 18:18	10/31/22 16:50	72-20-8	
Endrin aldehyde	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 16:50	7421-93-4	
Heptachlor	ND	ug/L	0.048	0.0096	1	10/19/22 18:18	10/31/22 16:50	76-44-8	
Heptachlor epoxide	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 16:50	1024-57-3	
Toxaphene	ND	ug/L	0.96	0.35	1	10/19/22 18:18	10/31/22 16:50	8001-35-2	
<b>Surrogates</b>									
Decachlorobiphenyl (S)	99	%	1-140		1	10/19/22 18:18	10/31/22 16:50	2051-24-3	
<b>200.8 Metals, Total ICMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Antimony	0.00036J	mg/L	0.0010	0.00013	1	10/19/22 08:00	10/20/22 13:55	7440-36-0	
Arsenic	0.00099J	mg/L	0.0010	0.00011	1	10/19/22 08:00	10/20/22 13:55	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	10/19/22 08:00	10/20/22 13:55	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	10/19/22 08:00	10/20/22 13:55	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	10/19/22 08:00	10/20/22 13:55	7440-47-3	
Copper	0.0020	mg/L	0.0010	0.00037	1	10/19/22 08:00	10/20/22 13:55	7440-50-8	
Lead	0.00033J	mg/L	0.0010	0.000080	1	10/19/22 08:00	10/20/22 13:55	7439-92-1	
Nickel	0.0028	mg/L	0.00050	0.00039	1	10/19/22 08:00	10/20/22 13:55	7440-02-0	
Selenium	0.00052J	mg/L	0.0010	0.00035	1	10/19/22 08:00	10/20/22 13:55	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	10/19/22 08:00	10/20/22 13:55	7440-22-4	

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### ANALYTICAL RESULTS

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 30-UPS-10172022 Lab ID: 50328663006 Collected: 10/17/22 12:25 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	ND	mg/L	0.0010	0.000073	1	10/19/22 08:00	10/20/22 13:55	7440-28-0	
Zinc	0.075	mg/L	0.0030	0.0010	1	10/19/22 08:00	10/20/22 13:55	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/23/22 19:34	10/24/22 12:00	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.6	1.7	1	10/20/22 15:49	10/28/22 21:49	83-32-9	
Acenaphthylene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:49	208-96-8	
Anthracene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:49	120-12-7	
Benzdine	ND	ug/L	48.1	5.8	1	10/20/22 15:49	10/28/22 21:49	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.6	1.6	1	10/20/22 15:49	10/28/22 21:49	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:49	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:49	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 21:49	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:49	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 21:49	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.6	3.6	1	10/20/22 15:49	10/28/22 21:49	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.2	2.9	1	10/20/22 15:49	10/28/22 21:49	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 21:49	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 21:49	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 21:49	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:49	91-58-7	
2-Chlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:49	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 21:49	7005-72-3	
Chrysene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:49	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:49	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 21:49	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 21:49	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 21:49	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.2	3.0	1	10/20/22 15:49	10/28/22 21:49	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:49	120-83-2	
Diethylphthalate	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 21:49	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:49	105-67-9	
Dimethylphthalate	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 21:49	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 21:49	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	48.1	7.8	1	10/20/22 15:49	10/28/22 21:49	534-52-1	
2,4-Dinitrophenol	ND	ug/L	48.1	5.0	1	10/20/22 15:49	10/28/22 21:49	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 21:49	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 21:49	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.6	7.1	1	10/20/22 15:49	10/28/22 21:49	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.6	2.0	1	10/20/22 15:49	10/28/22 21:49	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.8	4.0	1	10/20/22 15:49	10/28/22 21:49	117-81-7	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 30-UPS-10172022**      **Lab ID: 50328663006**      Collected: 10/17/22 12:25      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**625.1 MSSV**

Analytical Method: EPA 625.1      Preparation Method: EPA 625.1  
Pace Analytical Services - Indianapolis

Fluoranthene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:49	206-44-0	
Fluorene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 21:49	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.6	3.7	1	10/20/22 15:49	10/28/22 21:49	87-68-3	
Hexachlorobenzene	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 21:49	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.2	4.4	1	10/20/22 15:49	10/28/22 21:49	77-47-4	N2
Hexachloroethane	ND	ug/L	9.6	3.3	1	10/20/22 15:49	10/28/22 21:49	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 21:49	193-39-5	
Isophorone	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 21:49	78-59-1	
Naphthalene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 21:49	91-20-3	
Nitrobenzene	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 21:49	98-95-3	
2-Nitrophenol	ND	ug/L	9.6	4.0	1	10/20/22 15:49	10/28/22 21:49	88-75-5	
4-Nitrophenol	ND	ug/L	48.1	4.9	1	10/20/22 15:49	10/28/22 21:49	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.2	3.4	1	10/20/22 15:49	10/28/22 21:49	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 21:49	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 21:49	86-30-6	
Pentachlorophenol	ND	ug/L	48.1	6.5	1	10/20/22 15:49	10/28/22 21:49	87-86-5	
Phenanthrene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 21:49	85-01-8	
Phenol	ND	ug/L	9.6	1.2	1	10/20/22 15:49	10/28/22 21:49	108-95-2	
Pyrene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 21:49	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 21:49	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 21:49	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	5	%	9-74		1	10/20/22 15:49	10/28/22 21:49	367-12-4	H7,S0
Phenol-d5 (S)	4	%	8-424		1	10/20/22 15:49	10/28/22 21:49	4165-62-2	H7,S0
Nitrobenzene-d5 (S)	10	%	15-314		1	10/20/22 15:49	10/28/22 21:49	4165-60-0	H7,S0
2-Fluorobiphenyl (S)	14	%	32-92		1	10/20/22 15:49	10/28/22 21:49	321-60-8	H7,S0
2,4,6-Tribromophenol (S)	30	%	27-125		1	10/20/22 15:49	10/28/22 21:49	118-79-6	
p-Terphenyl-d14 (S)	24	%	8-146		1	10/20/22 15:49	10/28/22 21:49	1718-51-0	

**624.1 Volatile Organics**

Analytical Method: EPA 624.1  
Pace Analytical Services - Indianapolis

Acrolein	ND	ug/L	50.0	10	1		10/19/22 20:27	107-02-8	
Acrylonitrile	ND	ug/L	100	2.4	1		10/19/22 20:27	107-13-1	
Benzene	ND	ug/L	5.0	0.82	1		10/19/22 20:27	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.82	1		10/19/22 20:27	75-27-4	
Bromoform	ND	ug/L	5.0	0.73	1		10/19/22 20:27	75-25-2	
Bromomethane	ND	ug/L	5.0	0.44	1		10/19/22 20:27	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.68	1		10/19/22 20:27	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.95	1		10/19/22 20:27	108-90-7	
Chloroethane	ND	ug/L	5.0	0.63	1		10/19/22 20:27	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	2.7	1		10/19/22 20:27	110-75-8	
Chloroform	ND	ug/L	4.8	0.83	1		10/19/22 20:27	67-66-3	
Chloromethane	ND	ug/L	5.0	0.44	1		10/19/22 20:27	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.89	1		10/19/22 20:27	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.84	1		10/19/22 20:27	75-34-3	

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 30-UPS-10172022**      **Lab ID: 50328663006**      Collected: 10/17/22 12:25      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.85	1		10/19/22 20:27	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.56	1		10/19/22 20:27	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.72	1		10/19/22 20:27	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.79	1		10/19/22 20:27	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.86	1		10/19/22 20:27	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.92	1		10/19/22 20:27	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.95	1		10/19/22 20:27	100-41-4	
Methylene Chloride	ND	ug/L	5.0	0.70	1		10/19/22 20:27	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.92	1		10/19/22 20:27	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.75	1		10/19/22 20:27	127-18-4	
Toluene	ND	ug/L	5.0	0.86	1		10/19/22 20:27	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.74	1		10/19/22 20:27	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.88	1		10/19/22 20:27	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.80	1		10/19/22 20:27	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.52	1		10/19/22 20:27	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	100	%	91-114		1		10/19/22 20:27	1868-53-7	
4-Bromofluorobenzene (S)	101	%	85-120		1		10/19/22 20:27	460-00-4	
Toluene-d8 (S)	98	%	85-117		1		10/19/22 20:27	2037-26-5	

**335.4 Cyanide, Total**

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	ND	mg/L	0.0050	0.0018	1	10/20/22 08:00	10/20/22 14:05	57-12-5	
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**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 30-EFF-10172022**      **Lab ID: 50328663007**      Collected: 10/17/22 12:05      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>608.3 PCB</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
PCB-1016 (Aroclor 1016)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:36	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:36	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:36	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:36	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:36	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:36	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.096	0.049	1	10/19/22 18:18	10/25/22 17:36	11096-82-5	
<b>Surrogates</b>									
Tetrachloro-m-xylene (S)	52	%	1-123		1	10/19/22 18:18	10/25/22 17:36	877-09-8	
<b>608.3 Pesticides</b>									
Analytical Method: EPA 608.3 Preparation Method: EPA 608.3									
Pace Analytical Services - Indianapolis									
Aldrin	ND	ug/L	0.048	0.0087	1	10/19/22 18:18	10/31/22 17:02	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 17:02	319-84-6	
beta-BHC	ND	ug/L	0.048	0.013	1	10/19/22 18:18	10/31/22 17:02	319-85-7	
delta-BHC	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 17:02	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 17:02	58-89-9	
Chlordane (Technical)	ND	ug/L	0.48	0.26	1	10/19/22 18:18	10/31/22 17:02	57-74-9	
4,4'-DDD	ND	ug/L	0.096	0.023	1	10/19/22 18:18	10/31/22 17:02	72-54-8	
4,4'-DDE	ND	ug/L	0.096	0.017	1	10/19/22 18:18	10/31/22 17:02	72-55-9	
4,4'-DDT	ND	ug/L	0.096	0.035	1	10/19/22 18:18	10/31/22 17:02	50-29-3	
Dieldrin	ND	ug/L	0.096	0.021	1	10/19/22 18:18	10/31/22 17:02	60-57-1	
Endosulfan I	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 17:02	959-98-8	
Endosulfan II	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 17:02	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.096	0.020	1	10/19/22 18:18	10/31/22 17:02	1031-07-8	
Endrin	ND	ug/L	0.096	0.026	1	10/19/22 18:18	10/31/22 17:02	72-20-8	
Endrin aldehyde	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 17:02	7421-93-4	
Heptachlor	ND	ug/L	0.048	0.0096	1	10/19/22 18:18	10/31/22 17:02	76-44-8	
Heptachlor epoxide	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 17:02	1024-57-3	
Toxaphene	ND	ug/L	0.96	0.35	1	10/19/22 18:18	10/31/22 17:02	8001-35-2	
<b>Surrogates</b>									
Decachlorobiphenyl (S)	50	%	1-140		1	10/19/22 18:18	10/31/22 17:02	2051-24-3	
<b>200.8 Metals, Total ICMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Antimony	<b>0.0011</b>	mg/L	0.0010	0.00013	1	10/19/22 08:00	10/20/22 13:59	7440-36-0	
Arsenic	<b>0.0095</b>	mg/L	0.0010	0.00011	1	10/19/22 08:00	10/20/22 13:59	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	10/19/22 08:00	10/20/22 13:59	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	10/19/22 08:00	10/20/22 13:59	7440-43-9	
Chromium	<b>0.00067J</b>	mg/L	0.0020	0.00063	1	10/19/22 08:00	10/20/22 13:59	7440-47-3	
Copper	<b>0.0071</b>	mg/L	0.0010	0.00037	1	10/19/22 08:00	10/20/22 13:59	7440-50-8	
Lead	ND	mg/L	0.0010	0.000080	1	10/19/22 08:00	10/20/22 13:59	7439-92-1	
Nickel	<b>0.0046</b>	mg/L	0.00050	0.00039	1	10/19/22 08:00	10/20/22 13:59	7440-02-0	
Selenium	<b>0.092</b>	mg/L	0.0010	0.00035	1	10/19/22 08:00	10/20/22 13:59	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	10/19/22 08:00	10/20/22 13:59	7440-22-4	

**REPORT OF LABORATORY ANALYSIS**

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### ANALYTICAL RESULTS

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 30-EFF-10172022 Lab ID: 50328663007 Collected: 10/17/22 12:05 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	0.00026J	mg/L	0.0010	0.000073	1	10/19/22 08:00	10/20/22 13:59	7440-28-0	
Zinc	0.013	mg/L	0.0030	0.0010	1	10/19/22 08:00	10/20/22 13:59	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/23/22 19:34	10/24/22 12:03	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.6	1.7	1	10/20/22 15:49	10/28/22 22:05	83-32-9	
Acenaphthylene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:05	208-96-8	
Anthracene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:05	120-12-7	
Benzdine	ND	ug/L	48.1	5.8	1	10/20/22 15:49	10/28/22 22:05	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.6	1.6	1	10/20/22 15:49	10/28/22 22:05	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:05	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:05	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 22:05	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:05	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 22:05	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.6	3.6	1	10/20/22 15:49	10/28/22 22:05	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.2	2.9	1	10/20/22 15:49	10/28/22 22:05	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 22:05	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 22:05	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 22:05	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:05	91-58-7	
2-Chlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:05	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 22:05	7005-72-3	
Chrysene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:05	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:05	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 22:05	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 22:05	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 22:05	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.2	3.0	1	10/20/22 15:49	10/28/22 22:05	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:05	120-83-2	
Diethylphthalate	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 22:05	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:05	105-67-9	
Dimethylphthalate	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:05	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 22:05	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	48.1	7.8	1	10/20/22 15:49	10/28/22 22:05	534-52-1	
2,4-Dinitrophenol	ND	ug/L	48.1	5.0	1	10/20/22 15:49	10/28/22 22:05	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 22:05	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 22:05	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.6	7.1	1	10/20/22 15:49	10/28/22 22:05	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.6	2.0	1	10/20/22 15:49	10/28/22 22:05	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.8	4.0	1	10/20/22 15:49	10/28/22 22:05	117-81-7	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 30-EFF-10172022**      **Lab ID: 50328663007**      Collected: 10/17/22 12:05      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**625.1 MSSV**

Analytical Method: EPA 625.1      Preparation Method: EPA 625.1  
Pace Analytical Services - Indianapolis

Fluoranthene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:05	206-44-0	
Fluorene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 22:05	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.6	3.7	1	10/20/22 15:49	10/28/22 22:05	87-68-3	
Hexachlorobenzene	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 22:05	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.2	4.4	1	10/20/22 15:49	10/28/22 22:05	77-47-4	N2
Hexachloroethane	ND	ug/L	9.6	3.3	1	10/20/22 15:49	10/28/22 22:05	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:05	193-39-5	
Isophorone	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 22:05	78-59-1	
Naphthalene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 22:05	91-20-3	
Nitrobenzene	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 22:05	98-95-3	
2-Nitrophenol	ND	ug/L	9.6	4.0	1	10/20/22 15:49	10/28/22 22:05	88-75-5	
4-Nitrophenol	ND	ug/L	48.1	4.9	1	10/20/22 15:49	10/28/22 22:05	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.2	3.4	1	10/20/22 15:49	10/28/22 22:05	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 22:05	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 22:05	86-30-6	
Pentachlorophenol	ND	ug/L	48.1	6.5	1	10/20/22 15:49	10/28/22 22:05	87-86-5	
Phenanthrene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:05	85-01-8	
Phenol	ND	ug/L	9.6	1.2	1	10/20/22 15:49	10/28/22 22:05	108-95-2	
Pyrene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 22:05	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 22:05	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 22:05	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	40	%	9-74		1	10/20/22 15:49	10/28/22 22:05	367-12-4	
Phenol-d5 (S)	28	%	8-424		1	10/20/22 15:49	10/28/22 22:05	4165-62-2	
Nitrobenzene-d5 (S)	74	%	15-314		1	10/20/22 15:49	10/28/22 22:05	4165-60-0	
2-Fluorobiphenyl (S)	75	%	32-92		1	10/20/22 15:49	10/28/22 22:05	321-60-8	
2,4,6-Tribromophenol (S)	92	%	27-125		1	10/20/22 15:49	10/28/22 22:05	118-79-6	
p-Terphenyl-d14 (S)	79	%	8-146		1	10/20/22 15:49	10/28/22 22:05	1718-51-0	

**624.1 Volatile Organics**

Analytical Method: EPA 624.1  
Pace Analytical Services - Indianapolis

Acrolein	ND	ug/L	50.0	10	1		10/19/22 22:05	107-02-8	
Acrylonitrile	ND	ug/L	100	2.4	1		10/19/22 22:05	107-13-1	
Benzene	ND	ug/L	5.0	0.82	1		10/19/22 22:05	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.82	1		10/19/22 22:05	75-27-4	
Bromoform	ND	ug/L	5.0	0.73	1		10/19/22 22:05	75-25-2	
Bromomethane	ND	ug/L	5.0	0.44	1		10/19/22 22:05	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.68	1		10/19/22 22:05	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.95	1		10/19/22 22:05	108-90-7	
Chloroethane	ND	ug/L	5.0	0.63	1		10/19/22 22:05	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	2.7	1		10/19/22 22:05	110-75-8	
Chloroform	ND	ug/L	4.8	0.83	1		10/19/22 22:05	67-66-3	
Chloromethane	ND	ug/L	5.0	0.44	1		10/19/22 22:05	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.89	1		10/19/22 22:05	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.84	1		10/19/22 22:05	75-34-3	

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 30-EFF-10172022**      **Lab ID: 50328663007**      Collected: 10/17/22 12:05      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.85	1		10/19/22 22:05	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.56	1		10/19/22 22:05	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.72	1		10/19/22 22:05	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.79	1		10/19/22 22:05	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.86	1		10/19/22 22:05	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.92	1		10/19/22 22:05	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.95	1		10/19/22 22:05	100-41-4	
Methylene Chloride	ND	ug/L	5.0	0.70	1		10/19/22 22:05	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.92	1		10/19/22 22:05	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.75	1		10/19/22 22:05	127-18-4	
Toluene	ND	ug/L	5.0	0.86	1		10/19/22 22:05	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.74	1		10/19/22 22:05	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.88	1		10/19/22 22:05	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.80	1		10/19/22 22:05	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.52	1		10/19/22 22:05	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	103	%	91-114		1		10/19/22 22:05	1868-53-7	
4-Bromofluorobenzene (S)	102	%	85-120		1		10/19/22 22:05	460-00-4	
Toluene-d8 (S)	99	%	85-117		1		10/19/22 22:05	2037-26-5	
<b>335.4 Cyanide, Total</b>									
Analytical Method: EPA 335.4      Preparation Method: EPA 335.4									
Pace Analytical Services - Indianapolis									
Cyanide	<b>0.0040J</b>	mg/L	0.0050	0.0018	1	10/20/22 08:00	10/20/22 14:05	57-12-5	

**REPORT OF LABORATORY ANALYSIS**

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 30-DNS-10172022 Lab ID: 50328663008 Collected: 10/17/22 11:35 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				

**608.3 PCB**

Analytical Method: EPA 608.3 Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

PCB-1016 (Aroclor 1016)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:50	12674-11-2	
PCB-1221 (Aroclor 1221)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:50	11104-28-2	
PCB-1232 (Aroclor 1232)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:50	11141-16-5	
PCB-1242 (Aroclor 1242)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:50	53469-21-9	
PCB-1248 (Aroclor 1248)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:50	12672-29-6	
PCB-1254 (Aroclor 1254)	ND	ug/L	0.096	0.056	1	10/19/22 18:18	10/25/22 17:50	11097-69-1	
PCB-1260 (Aroclor 1260)	ND	ug/L	0.096	0.049	1	10/19/22 18:18	10/25/22 17:50	11096-82-5	

**Surrogates**

Tetrachloro-m-xylene (S)	45	%	1-123		1	10/19/22 18:18	10/25/22 17:50	877-09-8	
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**608.3 Pesticides**

Analytical Method: EPA 608.3 Preparation Method: EPA 608.3  
Pace Analytical Services - Indianapolis

Aldrin	ND	ug/L	0.048	0.0087	1	10/19/22 18:18	10/31/22 17:15	309-00-2	H7,L2
alpha-BHC	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 17:15	319-84-6	
beta-BHC	ND	ug/L	0.048	0.013	1	10/19/22 18:18	10/31/22 17:15	319-85-7	
delta-BHC	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 17:15	319-86-8	
gamma-BHC (Lindane)	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 17:15	58-89-9	
Chlordane (Technical)	ND	ug/L	0.48	0.26	1	10/19/22 18:18	10/31/22 17:15	57-74-9	
4,4'-DDD	ND	ug/L	0.096	0.023	1	10/19/22 18:18	10/31/22 17:15	72-54-8	
4,4'-DDE	ND	ug/L	0.096	0.017	1	10/19/22 18:18	10/31/22 17:15	72-55-9	
4,4'-DDT	ND	ug/L	0.096	0.035	1	10/19/22 18:18	10/31/22 17:15	50-29-3	
Dieldrin	ND	ug/L	0.096	0.021	1	10/19/22 18:18	10/31/22 17:15	60-57-1	
Endosulfan I	ND	ug/L	0.048	0.012	1	10/19/22 18:18	10/31/22 17:15	959-98-8	
Endosulfan II	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 17:15	33213-65-9	
Endosulfan sulfate	ND	ug/L	0.096	0.020	1	10/19/22 18:18	10/31/22 17:15	1031-07-8	
Endrin	ND	ug/L	0.096	0.026	1	10/19/22 18:18	10/31/22 17:15	72-20-8	
Endrin aldehyde	ND	ug/L	0.096	0.024	1	10/19/22 18:18	10/31/22 17:15	7421-93-4	
Heptachlor	ND	ug/L	0.048	0.0096	1	10/19/22 18:18	10/31/22 17:15	76-44-8	
Heptachlor epoxide	ND	ug/L	0.048	0.011	1	10/19/22 18:18	10/31/22 17:15	1024-57-3	
Toxaphene	ND	ug/L	0.96	0.35	1	10/19/22 18:18	10/31/22 17:15	8001-35-2	

**Surrogates**

Decachlorobiphenyl (S)	52	%	1-140		1	10/19/22 18:18	10/31/22 17:15	2051-24-3	
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**200.8 Metals, Total ICPMS**

Analytical Method: EPA 200.8 Preparation Method: EPA 200.8  
Pace Analytical Services - Indianapolis

Antimony	<b>0.00075J</b>	mg/L	0.0010	0.00013	1	10/19/22 08:00	10/20/22 14:03	7440-36-0	
Arsenic	<b>0.0046</b>	mg/L	0.0010	0.00011	1	10/19/22 08:00	10/20/22 14:03	7440-38-2	
Beryllium	ND	mg/L	0.00020	0.000033	1	10/19/22 08:00	10/20/22 14:03	7440-41-7	
Cadmium	ND	mg/L	0.00020	0.000034	1	10/19/22 08:00	10/20/22 14:03	7440-43-9	
Chromium	ND	mg/L	0.0020	0.00063	1	10/19/22 08:00	10/20/22 14:03	7440-47-3	
Copper	<b>0.0050</b>	mg/L	0.0010	0.00037	1	10/19/22 08:00	10/20/22 14:03	7440-50-8	
Lead	<b>0.00013J</b>	mg/L	0.0010	0.000080	1	10/19/22 08:00	10/20/22 14:03	7439-92-1	
Nickel	<b>0.0036</b>	mg/L	0.00050	0.00039	1	10/19/22 08:00	10/20/22 14:03	7440-02-0	
Selenium	<b>0.058</b>	mg/L	0.0010	0.00035	1	10/19/22 08:00	10/20/22 14:03	7782-49-2	
Silver	ND	mg/L	0.00050	0.000037	1	10/19/22 08:00	10/20/22 14:03	7440-22-4	

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### ANALYTICAL RESULTS

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Sample: 30-DNS-10172022 Lab ID: 50328663008 Collected: 10/17/22 11:35 Received: 10/18/22 12:20 Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>200.8 Metals, Total ICPMS</b>									
Analytical Method: EPA 200.8 Preparation Method: EPA 200.8									
Pace Analytical Services - Indianapolis									
Thallium	0.00012J	mg/L	0.0010	0.000073	1	10/19/22 08:00	10/20/22 14:03	7440-28-0	
Zinc	0.0073	mg/L	0.0030	0.0010	1	10/19/22 08:00	10/20/22 14:03	7440-66-6	
<b>245.1 Mercury</b>									
Analytical Method: EPA 245.1 Preparation Method: EPA 245.1									
Pace Analytical Services - Indianapolis									
Mercury	ND	mg/L	0.00020	0.00012	1	10/23/22 19:34	10/24/22 12:05	7439-97-6	
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1 Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Acenaphthene	ND	ug/L	9.6	1.7	1	10/20/22 15:49	10/28/22 22:22	83-32-9	
Acenaphthylene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:22	208-96-8	
Anthracene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:22	120-12-7	
Benzdine	ND	ug/L	48.1	5.8	1	10/20/22 15:49	10/28/22 22:22	92-87-5	
Benzo(a)anthracene	ND	ug/L	9.6	1.6	1	10/20/22 15:49	10/28/22 22:22	56-55-3	
Benzo(a)pyrene	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:22	50-32-8	
Benzo(b)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:22	205-99-2	
Benzo(g,h,i)perylene	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 22:22	191-24-2	
Benzo(k)fluoranthene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:22	207-08-9	
4-Bromophenylphenyl ether	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 22:22	101-55-3	
Butylbenzylphthalate	ND	ug/L	9.6	3.6	1	10/20/22 15:49	10/28/22 22:22	85-68-7	
4-Chloro-3-methylphenol	ND	ug/L	19.2	2.9	1	10/20/22 15:49	10/28/22 22:22	59-50-7	
bis(2-Chloroethoxy)methane	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 22:22	111-91-1	
bis(2-Chloroethyl) ether	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 22:22	111-44-4	
bis(2-Chloroisopropyl) ether	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 22:22	108-60-1	
2-Chloronaphthalene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:22	91-58-7	
2-Chlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:22	95-57-8	
4-Chlorophenylphenyl ether	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 22:22	7005-72-3	
Chrysene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:22	218-01-9	
Dibenz(a,h)anthracene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:22	53-70-3	
1,2-Dichlorobenzene	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 22:22	95-50-1	
1,3-Dichlorobenzene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 22:22	541-73-1	
1,4-Dichlorobenzene	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 22:22	106-46-7	
3,3'-Dichlorobenzidine	ND	ug/L	19.2	3.0	1	10/20/22 15:49	10/28/22 22:22	91-94-1	
2,4-Dichlorophenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:22	120-83-2	
Diethylphthalate	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 22:22	84-66-2	
2,4-Dimethylphenol	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:22	105-67-9	
Dimethylphthalate	ND	ug/L	9.6	2.2	1	10/20/22 15:49	10/28/22 22:22	131-11-3	
Di-n-butylphthalate	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 22:22	84-74-2	
4,6-Dinitro-2-methylphenol	ND	ug/L	48.1	7.8	1	10/20/22 15:49	10/28/22 22:22	534-52-1	
2,4-Dinitrophenol	ND	ug/L	48.1	5.0	1	10/20/22 15:49	10/28/22 22:22	51-28-5	
2,4-Dinitrotoluene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 22:22	121-14-2	
2,6-Dinitrotoluene	ND	ug/L	9.6	2.7	1	10/20/22 15:49	10/28/22 22:22	606-20-2	
Di-n-octylphthalate	ND	ug/L	9.6	7.1	1	10/20/22 15:49	10/28/22 22:22	117-84-0	
1,2-Diphenylhydrazine	ND	ug/L	9.6	2.0	1	10/20/22 15:49	10/28/22 22:22	122-66-7	N2
bis(2-Ethylhexyl)phthalate	ND	ug/L	4.8	4.0	1	10/20/22 15:49	10/28/22 22:22	117-81-7	

### REPORT OF LABORATORY ANALYSIS

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 30-DNS-10172022**      **Lab ID: 50328663008**      Collected: 10/17/22 11:35      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report			Prepared	Analyzed	CAS No.	Qual
			Limit	MDL	DF				
<b>625.1 MSSV</b>									
Analytical Method: EPA 625.1    Preparation Method: EPA 625.1									
Pace Analytical Services - Indianapolis									
Fluoranthene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:22	206-44-0	
Fluorene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 22:22	86-73-7	
Hexachloro-1,3-butadiene	ND	ug/L	9.6	3.7	1	10/20/22 15:49	10/28/22 22:22	87-68-3	
Hexachlorobenzene	ND	ug/L	9.6	3.2	1	10/20/22 15:49	10/28/22 22:22	118-74-1	
Hexachlorocyclopentadiene	ND	ug/L	19.2	4.4	1	10/20/22 15:49	10/28/22 22:22	77-47-4	N2
Hexachloroethane	ND	ug/L	9.6	3.3	1	10/20/22 15:49	10/28/22 22:22	67-72-1	N2
Indeno(1,2,3-cd)pyrene	ND	ug/L	9.6	2.5	1	10/20/22 15:49	10/28/22 22:22	193-39-5	
Isophorone	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 22:22	78-59-1	
Naphthalene	ND	ug/L	9.6	2.3	1	10/20/22 15:49	10/28/22 22:22	91-20-3	
Nitrobenzene	ND	ug/L	9.6	3.0	1	10/20/22 15:49	10/28/22 22:22	98-95-3	
2-Nitrophenol	ND	ug/L	9.6	4.0	1	10/20/22 15:49	10/28/22 22:22	88-75-5	
4-Nitrophenol	ND	ug/L	48.1	4.9	1	10/20/22 15:49	10/28/22 22:22	100-02-7	
N-Nitrosodimethylamine	ND	ug/L	19.2	3.4	1	10/20/22 15:49	10/28/22 22:22	62-75-9	
N-Nitroso-di-n-propylamine	ND	ug/L	9.6	2.8	1	10/20/22 15:49	10/28/22 22:22	621-64-7	
N-Nitrosodiphenylamine	ND	ug/L	9.6	2.1	1	10/20/22 15:49	10/28/22 22:22	86-30-6	
Pentachlorophenol	ND	ug/L	48.1	6.5	1	10/20/22 15:49	10/28/22 22:22	87-86-5	
Phenanthrene	ND	ug/L	9.6	1.8	1	10/20/22 15:49	10/28/22 22:22	85-01-8	
Phenol	ND	ug/L	9.6	1.2	1	10/20/22 15:49	10/28/22 22:22	108-95-2	
Pyrene	ND	ug/L	9.6	1.9	1	10/20/22 15:49	10/28/22 22:22	129-00-0	
1,2,4-Trichlorobenzene	ND	ug/L	9.6	3.5	1	10/20/22 15:49	10/28/22 22:22	120-82-1	
2,4,6-Trichlorophenol	ND	ug/L	9.6	2.6	1	10/20/22 15:49	10/28/22 22:22	88-06-2	
<b>Surrogates</b>									
2-Fluorophenol (S)	41	%	9-74		1	10/20/22 15:49	10/28/22 22:22	367-12-4	
Phenol-d5 (S)	29	%	8-424		1	10/20/22 15:49	10/28/22 22:22	4165-62-2	
Nitrobenzene-d5 (S)	77	%	15-314		1	10/20/22 15:49	10/28/22 22:22	4165-60-0	
2-Fluorobiphenyl (S)	78	%	32-92		1	10/20/22 15:49	10/28/22 22:22	321-60-8	
2,4,6-Tribromophenol (S)	93	%	27-125		1	10/20/22 15:49	10/28/22 22:22	118-79-6	
p-Terphenyl-d14 (S)	82	%	8-146		1	10/20/22 15:49	10/28/22 22:22	1718-51-0	

**624.1 Volatile Organics**

Analytical Method: EPA 624.1

Pace Analytical Services - Indianapolis

Acrolein	ND	ug/L	50.0	10	1		10/19/22 21:00	107-02-8	
Acrylonitrile	ND	ug/L	100	2.4	1		10/19/22 21:00	107-13-1	
Benzene	ND	ug/L	5.0	0.82	1		10/19/22 21:00	71-43-2	
Bromodichloromethane	ND	ug/L	5.0	0.82	1		10/19/22 21:00	75-27-4	
Bromoform	ND	ug/L	5.0	0.73	1		10/19/22 21:00	75-25-2	
Bromomethane	ND	ug/L	5.0	0.44	1		10/19/22 21:00	74-83-9	
Carbon tetrachloride	ND	ug/L	5.0	0.68	1		10/19/22 21:00	56-23-5	
Chlorobenzene	ND	ug/L	5.0	0.95	1		10/19/22 21:00	108-90-7	
Chloroethane	ND	ug/L	5.0	0.63	1		10/19/22 21:00	75-00-3	
2-Chloroethylvinyl ether	ND	ug/L	50.0	2.7	1		10/19/22 21:00	110-75-8	
Chloroform	ND	ug/L	4.8	0.83	1		10/19/22 21:00	67-66-3	
Chloromethane	ND	ug/L	5.0	0.44	1		10/19/22 21:00	74-87-3	
Dibromochloromethane	ND	ug/L	5.0	0.89	1		10/19/22 21:00	124-48-1	
1,1-Dichloroethane	ND	ug/L	5.0	0.84	1		10/19/22 21:00	75-34-3	

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**ANALYTICAL RESULTS**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

**Sample: 30-DNS-10172022**      **Lab ID: 50328663008**      Collected: 10/17/22 11:35      Received: 10/18/22 12:20      Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>624.1 Volatile Organics</b>									
Analytical Method: EPA 624.1									
Pace Analytical Services - Indianapolis									
1,2-Dichloroethane	ND	ug/L	5.0	0.85	1		10/19/22 21:00	107-06-2	
1,1-Dichloroethene	ND	ug/L	5.0	0.56	1		10/19/22 21:00	75-35-4	
trans-1,2-Dichloroethene	ND	ug/L	4.8	0.72	1		10/19/22 21:00	156-60-5	
1,2-Dichloropropane	ND	ug/L	5.0	0.79	1		10/19/22 21:00	78-87-5	
cis-1,3-Dichloropropene	ND	ug/L	5.0	0.86	1		10/19/22 21:00	10061-01-5	
trans-1,3-Dichloropropene	ND	ug/L	5.0	0.92	1		10/19/22 21:00	10061-02-6	
Ethylbenzene	ND	ug/L	5.0	0.95	1		10/19/22 21:00	100-41-4	
Methylene Chloride	ND	ug/L	5.0	0.70	1		10/19/22 21:00	75-09-2	
1,1,2,2-Tetrachloroethane	ND	ug/L	5.0	0.92	1		10/19/22 21:00	79-34-5	
Tetrachloroethene	ND	ug/L	5.0	0.75	1		10/19/22 21:00	127-18-4	
Toluene	ND	ug/L	5.0	0.86	1		10/19/22 21:00	108-88-3	
1,1,1-Trichloroethane	ND	ug/L	5.0	0.74	1		10/19/22 21:00	71-55-6	
1,1,2-Trichloroethane	ND	ug/L	5.0	0.88	1		10/19/22 21:00	79-00-5	
Trichloroethene	ND	ug/L	5.0	0.80	1		10/19/22 21:00	79-01-6	
Vinyl chloride	ND	ug/L	2.0	0.52	1		10/19/22 21:00	75-01-4	
<b>Surrogates</b>									
Dibromofluoromethane (S)	101	%	91-114		1		10/19/22 21:00	1868-53-7	
4-Bromofluorobenzene (S)	102	%	85-120		1		10/19/22 21:00	460-00-4	
Toluene-d8 (S)	100	%	85-117		1		10/19/22 21:00	2037-26-5	

**335.4 Cyanide, Total**

Analytical Method: EPA 335.4 Preparation Method: EPA 335.4

Pace Analytical Services - Indianapolis

Cyanide	<b>0.0026J</b>	mg/L	0.0050	0.0018	1	10/20/22 08:00	10/20/22 14:07	57-12-5	
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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

QC Batch:	702185	Analysis Method:	EPA 245.1
QC Batch Method:	EPA 245.1	Analysis Description:	245.1 Mercury
		Laboratory:	Pace Analytical Services - Indianapolis

Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

METHOD BLANK: 3228245 Matrix: Water

Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Mercury	mg/L	ND	0.00020	0.00012	10/24/22 10:59	

LABORATORY CONTROL SAMPLE: 3228246

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mercury	mg/L	0.005	0.0051	102	85-115	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3228247 3228248

Parameter	Units	50328644003 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mercury	mg/L	<0.20 ug/L	0.005	0.005	0.0051	0.0051	101	102	70-130	0	20	

MATRIX SPIKE SAMPLE: 3228249

Parameter	Units	50328663008 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Mercury	mg/L		ND	0.005	0.0050	100	70-130

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

QC Batch: 701458 Analysis Method: EPA 200.8  
QC Batch Method: EPA 200.8 Analysis Description: 200.8 MET  
Laboratory: Pace Analytical Services - Indianapolis  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

METHOD BLANK: 3224715 Matrix: Water  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Antimony	mg/L	ND	0.0010	0.00013	10/20/22 13:19	
Arsenic	mg/L	ND	0.0010	0.00011	10/20/22 13:19	
Beryllium	mg/L	ND	0.00020	0.000033	10/20/22 13:19	
Cadmium	mg/L	ND	0.00020	0.000034	10/20/22 13:19	
Chromium	mg/L	ND	0.0020	0.00063	10/20/22 13:19	
Copper	mg/L	ND	0.0010	0.00037	10/20/22 13:19	
Lead	mg/L	ND	0.0010	0.000080	10/20/22 13:19	
Nickel	mg/L	ND	0.00050	0.00039	10/20/22 13:19	
Selenium	mg/L	ND	0.0010	0.00035	10/20/22 13:19	
Silver	mg/L	ND	0.00050	0.00037	10/20/22 13:19	
Thallium	mg/L	ND	0.0010	0.000073	10/20/22 13:19	
Zinc	mg/L	ND	0.0030	0.0010	10/20/22 13:19	

LABORATORY CONTROL SAMPLE: 3224716

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Antimony	mg/L	0.04	0.043	107	85-115	
Arsenic	mg/L	0.04	0.040	100	85-115	
Beryllium	mg/L	0.04	0.042	104	85-115	
Cadmium	mg/L	0.04	0.040	99	85-115	
Chromium	mg/L	0.04	0.042	105	85-115	
Copper	mg/L	0.04	0.039	98	85-115	
Lead	mg/L	0.04	0.041	103	85-115	
Nickel	mg/L	0.04	0.039	99	85-115	
Selenium	mg/L	0.04	0.040	101	85-115	
Silver	mg/L	0.04	0.042	104	85-115	
Thallium	mg/L	0.04	0.042	106	85-115	
Zinc	mg/L	0.04	0.039	99	85-115	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3224717 3224718

Parameter	Units	MS		MSD		MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual	
		50328607001 Result	Spike Conc.	Spike Conc.	Result							Result
Antimony	mg/L	ND	0.04	0.04	0.045	0.045	111	111	70-130	0	20	
Arsenic	mg/L	ND	0.04	0.04	0.040	0.040	98	98	70-130	0	20	
Beryllium	mg/L	ND	0.04	0.04	0.039	0.039	97	97	70-130	0	20	

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3224717												3224718	
Parameter	Units	50328607001 Result	MS	MSD	MS	MSD	MS	MSD	% Rec	Max	Qual		
			Spike Conc.	Spike Conc.	Result	Result	% Rec	% Rec	Limits	RPD			
Cadmium	mg/L	ND	0.04	0.04	0.038	0.038	96	96	70-130	0	20		
Chromium	mg/L	ND	0.04	0.04	0.041	0.040	101	101	70-130	1	20		
Copper	mg/L	ND	0.04	0.04	0.036	0.036	89	90	70-130	0	20		
Lead	mg/L	ND	0.04	0.04	0.041	0.041	102	103	70-130	0	20		
Nickel	mg/L	0.89 ug/L	0.04	0.04	0.036	0.036	88	87	70-130	1	20		
Selenium	mg/L	ND	0.04	0.04	0.038	0.041	94	102	70-130	8	20		
Silver	mg/L	ND	0.04	0.04	0.039	0.039	98	97	70-130	0	20		
Thallium	mg/L	ND	0.04	0.04	0.043	0.043	107	107	70-130	0	20		
Zinc	mg/L	ND	0.04	0.04	0.038	0.039	93	93	70-130	1	20		

MATRIX SPIKE SAMPLE: 3224719		50328610001	Spike	MS	MS	% Rec	Qualifiers
Parameter	Units	Result	Conc.	Result	% Rec	Limits	
Antimony	mg/L	0.00030J	0.04	0.045	111	70-130	
Arsenic	mg/L	0.0049	0.04	0.045	101	70-130	
Beryllium	mg/L	ND	0.04	0.040	100	70-130	
Cadmium	mg/L	ND	0.04	0.038	95	70-130	
Chromium	mg/L	0.00083J	0.04	0.040	97	70-130	
Copper	mg/L	0.0011	0.04	0.036	88	70-130	
Lead	mg/L	0.00032J	0.04	0.041	103	70-130	
Nickel	mg/L	0.0030	0.04	0.038	87	70-130	
Selenium	mg/L	0.042	0.04	0.081	99	70-130	
Silver	mg/L	ND	0.04	0.039	96	70-130	
Thallium	mg/L	ND	0.04	0.043	106	70-130	
Zinc	mg/L	0.032	0.04	0.068	89	70-130	

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

QC Batch: 701585 Analysis Method: EPA 624.1  
QC Batch Method: EPA 624.1 Analysis Description: 624.1 MSV  
Laboratory: Pace Analytical Services - Indianapolis  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

METHOD BLANK: 3225176 Matrix: Water  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
1,1,1-Trichloroethane	ug/L	ND	5.0	0.74	10/19/22 12:53	
1,1,2,2-Tetrachloroethane	ug/L	ND	5.0	0.92	10/19/22 12:53	
1,1,2-Trichloroethane	ug/L	ND	5.0	0.88	10/19/22 12:53	
1,1-Dichloroethane	ug/L	ND	5.0	0.84	10/19/22 12:53	
1,1-Dichloroethene	ug/L	ND	5.0	0.56	10/19/22 12:53	
1,2-Dichloroethane	ug/L	ND	5.0	0.85	10/19/22 12:53	
1,2-Dichloropropane	ug/L	ND	5.0	0.79	10/19/22 12:53	
2-Chloroethylvinyl ether	ug/L	ND	50.0	2.7	10/19/22 12:53	
Acrolein	ug/L	ND	50.0	10	10/19/22 12:53	
Acrylonitrile	ug/L	ND	100	2.4	10/19/22 12:53	
Benzene	ug/L	ND	5.0	0.82	10/19/22 12:53	
Bromodichloromethane	ug/L	ND	5.0	0.82	10/19/22 12:53	
Bromoform	ug/L	ND	5.0	0.73	10/19/22 12:53	
Bromomethane	ug/L	ND	5.0	0.44	10/19/22 12:53	
Carbon tetrachloride	ug/L	ND	5.0	0.68	10/19/22 12:53	
Chlorobenzene	ug/L	ND	5.0	0.95	10/19/22 12:53	
Chloroethane	ug/L	ND	5.0	0.63	10/19/22 12:53	
Chloroform	ug/L	ND	4.8	0.83	10/19/22 12:53	
Chloromethane	ug/L	ND	5.0	0.44	10/19/22 12:53	
cis-1,3-Dichloropropene	ug/L	ND	5.0	0.86	10/19/22 12:53	
Dibromochloromethane	ug/L	ND	5.0	0.89	10/19/22 12:53	
Ethylbenzene	ug/L	ND	5.0	0.95	10/19/22 12:53	
Methylene Chloride	ug/L	ND	5.0	0.70	10/19/22 12:53	
Tetrachloroethene	ug/L	ND	5.0	0.75	10/19/22 12:53	
Toluene	ug/L	ND	5.0	0.86	10/19/22 12:53	
trans-1,2-Dichloroethene	ug/L	ND	4.8	0.72	10/19/22 12:53	
trans-1,3-Dichloropropene	ug/L	ND	5.0	0.92	10/19/22 12:53	
Trichloroethene	ug/L	ND	5.0	0.80	10/19/22 12:53	
Vinyl chloride	ug/L	ND	2.0	0.52	10/19/22 12:53	
4-Bromofluorobenzene (S)	%	101	85-120		10/19/22 12:53	
Dibromofluoromethane (S)	%	102	91-114		10/19/22 12:53	
Toluene-d8 (S)	%	99	85-117		10/19/22 12:53	

LABORATORY CONTROL SAMPLE: 3225177

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
1,1,1-Trichloroethane	ug/L	20	23.5	117	70-130	

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

LABORATORY CONTROL SAMPLE: 3225177

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
1,1,2,2-Tetrachloroethane	ug/L	20	22.4	112	60-140	
1,1,2-Trichloroethane	ug/L	20	21.8	109	70-130	
1,1-Dichloroethane	ug/L	20	22.1	110	70-130	
1,1-Dichloroethene	ug/L	20	22.1	111	50-150	
1,2-Dichloroethane	ug/L	20	23.8	119	70-130	
1,2-Dichloropropane	ug/L	20	21.9	109	35-165	
2-Chloroethylvinyl ether	ug/L	100	103	103	1-225	
Acrolein	ug/L	400	421	105	60-140	
Acrylonitrile	ug/L	100	117	117	60-140	
Benzene	ug/L	20	22.2	111	65-135	
Bromodichloromethane	ug/L	20	23.5	117	65-135	
Bromoform	ug/L	20	22.7	113	70-130	
Bromomethane	ug/L	20	16.5	82	15-185	
Carbon tetrachloride	ug/L	20	21.3	107	70-130	
Chlorobenzene	ug/L	20	22.1	111	65-135	
Chloroethane	ug/L	20	18.5	93	40-160	
Chloroform	ug/L	20	22.7	113	70-135	
Chloromethane	ug/L	20	19.2	96	1-205	
cis-1,3-Dichloropropene	ug/L	20	22.6	113	25-175	
Dibromochloromethane	ug/L	20	23.1	116	70-135	
Ethylbenzene	ug/L	20	21.7	108	60-140	
Methylene Chloride	ug/L	20	17.7	89	60-140	
Tetrachloroethene	ug/L	20	21.6	108	70-130	
Toluene	ug/L	20	21.0	105	70-130	
trans-1,2-Dichloroethene	ug/L	20	21.5	108	70-130	
trans-1,3-Dichloropropene	ug/L	20	21.4	107	50-150	
Trichloroethene	ug/L	20	24.2	121	65-135	
Vinyl chloride	ug/L	20	19.1	95	5-195	
4-Bromofluorobenzene (S)	%			101	85-120	
Dibromofluoromethane (S)	%			99	91-114	
Toluene-d8 (S)	%			96	85-117	

MATRIX SPIKE SAMPLE: 3225178

Parameter	Units	50328663007 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
1,1,1-Trichloroethane	ug/L	ND	20	29.2	146	52-162	
1,1,2,2-Tetrachloroethane	ug/L	ND	20	25.2	126	46-157	
1,1,2-Trichloroethane	ug/L	ND	20	26.4	132	52-150	
1,1-Dichloroethane	ug/L	ND	20	26.9	135	59-155	
1,1-Dichloroethene	ug/L	ND	20	28.8	144	1-234	
1,2-Dichloroethane	ug/L	ND	20	28.8	144	49-155	
1,2-Dichloropropane	ug/L	ND	20	26.2	131	1-210	
2-Chloroethylvinyl ether	ug/L	ND	100	123	123	1-305	
Acrolein	ug/L	ND	400	260	65	40-160	
Acrylonitrile	ug/L	ND	100	138	138	40-160	

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

MATRIX SPIKE SAMPLE: 3225178		50328663007	Spike	MS	MS	% Rec	
Parameter	Units	Result	Conc.	Result	% Rec	Limits	Qualifiers
Benzene	ug/L	ND	20	27.2	136	37-151	
Bromodichloromethane	ug/L	ND	20	28.4	142	35-155	
Bromoform	ug/L	ND	20	26.5	132	45-169	
Bromomethane	ug/L	ND	20	16.5	83	1-242	
Carbon tetrachloride	ug/L	ND	20	27.3	137	70-140	
Chlorobenzene	ug/L	ND	20	26.8	134	37-160	
Chloroethane	ug/L	ND	20	25.2	126	14-230	
Chloroform	ug/L	ND	20	27.6	138	51-138	
Chloromethane	ug/L	ND	20	27.9	139	1-273	
cis-1,3-Dichloropropene	ug/L	ND	20	27.1	135	1-227	
Dibromochloromethane	ug/L	ND	20	27.9	140	53-149	
Ethylbenzene	ug/L	ND	20	26.6	133	37-162	
Methylene Chloride	ug/L	ND	20	19.0	95	1-221	
Tetrachloroethene	ug/L	ND	20	27.2	136	64-148	
Toluene	ug/L	ND	20	25.9	129	47-150	
trans-1,2-Dichloroethene	ug/L	ND	20	27.3	137	54-156	
trans-1,3-Dichloropropene	ug/L	ND	20	25.5	128	17-183	
Trichloroethene	ug/L	ND	20	31.5	157	70-157	
Vinyl chloride	ug/L	ND	20	26.3	132	1-251	
4-Bromofluorobenzene (S)	%				103	85-120	
Dibromofluoromethane (S)	%				99	91-114	
Toluene-d8 (S)	%				98	85-117	

SAMPLE DUPLICATE: 3225179

Parameter	Units	50328663008	Dup	RPD	Max	Qualifiers
		Result	Result		RPD	
1,1,1-Trichloroethane	ug/L	ND	ND		36	
1,1,2,2-Tetrachloroethane	ug/L	ND	ND		61	
1,1,2-Trichloroethane	ug/L	ND	ND		45	
1,1-Dichloroethane	ug/L	ND	ND		40	
1,1-Dichloroethene	ug/L	ND	ND		32	
1,2-Dichloroethane	ug/L	ND	ND		49	
1,2-Dichloropropane	ug/L	ND	ND		55	
2-Chloroethylvinyl ether	ug/L	ND	ND		71	
Acrolein	ug/L	ND	ND		60	
Acrylonitrile	ug/L	ND	ND		60	
Benzene	ug/L	ND	ND		61	
Bromodichloromethane	ug/L	ND	ND		56	
Bromoform	ug/L	ND	ND		42	
Bromomethane	ug/L	ND	ND		61	
Carbon tetrachloride	ug/L	ND	ND		41	
Chlorobenzene	ug/L	ND	ND		53	
Chloroethane	ug/L	ND	ND		78	
Chloroform	ug/L	ND	ND		54	
Chloromethane	ug/L	ND	ND		60	

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

SAMPLE DUPLICATE: 3225179

Parameter	Units	50328663008 Result	Dup Result	RPD	Max RPD	Qualifiers
cis-1,3-Dichloropropene	ug/L	ND	ND		58	
Dibromochloromethane	ug/L	ND	ND		50	
Ethylbenzene	ug/L	ND	ND		63	
Methylene Chloride	ug/L	ND	ND		28	
Tetrachloroethene	ug/L	ND	ND		39	
Toluene	ug/L	ND	ND		41	
trans-1,2-Dichloroethene	ug/L	ND	ND		45	
trans-1,3-Dichloropropene	ug/L	ND	ND		86	
Trichloroethene	ug/L	ND	ND		48	
Vinyl chloride	ug/L	ND	ND		66	
4-Bromofluorobenzene (S)	%.	102	102			
Dibromofluoromethane (S)	%.	101	100			
Toluene-d8 (S)	%.	100	98			

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

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QC Batch:	701642	Analysis Method:	EPA 608.3
QC Batch Method:	EPA 608.3	Analysis Description:	608.3 PCB
		Laboratory:	Pace Analytical Services - Indianapolis

Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

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METHOD BLANK: 3225463 Matrix: Water  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
PCB-1016 (Aroclor 1016)	ug/L	ND	0.10	0.058	10/25/22 14:25	
PCB-1221 (Aroclor 1221)	ug/L	ND	0.10	0.058	10/25/22 14:25	
PCB-1232 (Aroclor 1232)	ug/L	ND	0.10	0.058	10/25/22 14:25	
PCB-1242 (Aroclor 1242)	ug/L	ND	0.10	0.058	10/25/22 14:25	
PCB-1248 (Aroclor 1248)	ug/L	ND	0.10	0.058	10/25/22 14:25	
PCB-1254 (Aroclor 1254)	ug/L	ND	0.10	0.058	10/25/22 14:25	
PCB-1260 (Aroclor 1260)	ug/L	ND	0.10	0.051	10/25/22 14:25	
Tetrachloro-m-xylene (S)	%	38	1-123		10/25/22 14:25	

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LABORATORY CONTROL SAMPLE: 3225464

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
PCB-1016 (Aroclor 1016)	ug/L	0.5	0.41	82	50-140	
PCB-1260 (Aroclor 1260)	ug/L	0.5	0.35	70	8-140	
Tetrachloro-m-xylene (S)	%			40	1-123	

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MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3225465 3225466

Parameter	Units	60413012001		MSD		MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	Max RPD	Qual
		Result	Conc.	Spike Conc.	Spike Conc.							
PCB-1016 (Aroclor 1016)	ug/L	ND	1	1	0.90	0.79	90	79	50-140	13	36	
PCB-1260 (Aroclor 1260)	ug/L	ND	1	1	0.66	0.63	66	63	8-140	5	38	
Tetrachloro-m-xylene (S)	%						74	72	1-123			

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

QC Batch: 701644 Analysis Method: EPA 608.3  
QC Batch Method: EPA 608.3 Analysis Description: 608.3 Pesticides  
Laboratory: Pace Analytical Services - Indianapolis  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

METHOD BLANK: 3225471 Matrix: Water  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
4,4'-DDD	ug/L	ND	0.10	0.024	10/25/22 14:37	
4,4'-DDE	ug/L	ND	0.10	0.018	10/25/22 14:37	
4,4'-DDT	ug/L	ND	0.10	0.036	10/25/22 14:37	
Aldrin	ug/L	ND	0.050	0.0090	10/25/22 14:37	
alpha-BHC	ug/L	ND	0.050	0.011	10/25/22 14:37	
beta-BHC	ug/L	ND	0.050	0.014	10/25/22 14:37	
Chlordane (Technical)	ug/L	ND	0.50	0.27	10/25/22 14:37	
delta-BHC	ug/L	ND	0.050	0.013	10/25/22 14:37	
Dieldrin	ug/L	ND	0.10	0.022	10/25/22 14:37	
Endosulfan I	ug/L	ND	0.050	0.012	10/25/22 14:37	
Endosulfan II	ug/L	ND	0.10	0.025	10/25/22 14:37	
Endosulfan sulfate	ug/L	ND	0.10	0.021	10/25/22 14:37	
Endrin	ug/L	ND	0.10	0.027	10/25/22 14:37	
Endrin aldehyde	ug/L	ND	0.10	0.025	10/25/22 14:37	
gamma-BHC (Lindane)	ug/L	ND	0.050	0.012	10/25/22 14:37	
Heptachlor	ug/L	ND	0.050	0.010	10/25/22 14:37	
Heptachlor epoxide	ug/L	ND	0.050	0.011	10/25/22 14:37	
Toxaphene	ug/L	ND	1.0	0.36	10/25/22 14:37	
Decachlorobiphenyl (S)	%	60	1-140		10/25/22 14:37	

LABORATORY CONTROL SAMPLE: 3225472

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
4,4'-DDD	ug/L	0.2	0.21	107	31-141	
4,4'-DDE	ug/L	0.2	0.14	72	30-145	
4,4'-DDT	ug/L	0.2	0.19	96	25-160	
Aldrin	ug/L	0.1	0.025J	25	42-140	L2
alpha-BHC	ug/L	0.1	0.10	100	37-140	
beta-BHC	ug/L	0.1	0.12	115	17-147	
delta-BHC	ug/L	0.1	0.059	59	19-140	
Dieldrin	ug/L	0.2	0.21	105	36-146	
Endosulfan I	ug/L	0.1	0.10	102	45-153	
Endosulfan II	ug/L	0.2	0.21	106	1-202	
Endosulfan sulfate	ug/L	0.2	0.19	96	26-144	
Endrin	ug/L	0.2	0.21	107	30-147	
Endrin aldehyde	ug/L	0.2	0.21	107	42-161	
gamma-BHC (Lindane)	ug/L	0.1	0.11	110	32-140	

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

LABORATORY CONTROL SAMPLE: 3225472

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Heptachlor	ug/L	0.1	0.039J	39	34-140	
Heptachlor epoxide	ug/L	0.1	0.10	101	37-142	
Decachlorobiphenyl (S)	%.			56	1-140	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3225473 3225474

Parameter	Units	MS		MSD		MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
		50328648001 Result	Spike Conc.	Spike Conc.	Result						
4,4'-DDD	ug/L	<0.10	0.4	0.4	0.39	0.35	100	88	31-141	12	39
4,4'-DDE	ug/L	<0.10	0.4	0.4	0.38	0.30	96	75	30-145	24	35
4,4'-DDT	ug/L	<0.10	0.4	0.4	0.39	0.32	99	80	25-160	22	42
Aldrin	ug/L	<0.050	0.2	0.2	0.19	0.093J	96	47	42-140		35
alpha-BHC	ug/L	<0.050	0.2	0.2	0.18	0.18	93	90	37-140	3	36
beta-BHC	ug/L	<0.050	0.2	0.2	0.21	0.20	106	101	17-147	5	44
delta-BHC	ug/L	<0.050	0.2	0.2	0.10	0.095J	52	48	19-140		52
Dieldrin	ug/L	<0.10	0.4	0.4	0.38	0.35	97	87	36-146	11	49
Endosulfan I	ug/L	<0.050	0.2	0.2	0.19	0.18	98	92	45-153	7	28
Endosulfan II	ug/L	<0.10	0.4	0.4	0.37	0.35	92	87	1-202	6	53
Endosulfan sulfate	ug/L	<0.10	0.4	0.4	0.33	0.32	84	81	26-144	4	38
Endrin	ug/L	<0.10	0.4	0.4	0.40	0.38	100	95	30-147	6	48
Endrin aldehyde	ug/L	<0.10	0.4	0.4	0.43	0.43	109	108	1-179	1	30
gamma-BHC (Lindane)	ug/L	<0.050	0.2	0.2	0.20	0.19	99	95	32-140	4	35
Heptachlor	ug/L	<0.050	0.2	0.2	0.19	0.13	97	65	34-140	40	43
Heptachlor epoxide	ug/L	<0.050	0.2	0.2	0.20	0.19	101	94	37-142	7	26
Decachlorobiphenyl (S)	%.						57	46	1-140		

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**REPORT OF LABORATORY ANALYSIS**

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

QC Batch: 701847 Analysis Method: EPA 625.1  
QC Batch Method: EPA 625.1 Analysis Description: 625.1 MSS  
Laboratory: Pace Analytical Services - Indianapolis  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

METHOD BLANK: 3226363 Matrix: Water  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
1,2,4-Trichlorobenzene	ug/L	ND	10.0	3.7	10/28/22 16:35	
1,2-Dichlorobenzene	ug/L	ND	10.0	2.7	10/28/22 16:35	
1,2-Diphenylhydrazine	ug/L	ND	10.0	2.1	10/28/22 16:35	N2
1,3-Dichlorobenzene	ug/L	ND	10.0	2.9	10/28/22 16:35	
1,4-Dichlorobenzene	ug/L	ND	10.0	2.9	10/28/22 16:35	
2,4,6-Trichlorophenol	ug/L	ND	10.0	2.8	10/28/22 16:35	
2,4-Dichlorophenol	ug/L	ND	10.0	2.3	10/28/22 16:35	
2,4-Dimethylphenol	ug/L	ND	10.0	2.3	10/28/22 16:35	
2,4-Dinitrophenol	ug/L	ND	50.0	5.2	10/28/22 16:35	
2,4-Dinitrotoluene	ug/L	ND	10.0	2.4	10/28/22 16:35	
2,6-Dinitrotoluene	ug/L	ND	10.0	2.9	10/28/22 16:35	
2-Chloronaphthalene	ug/L	ND	10.0	2.6	10/28/22 16:35	
2-Chlorophenol	ug/L	ND	10.0	2.3	10/28/22 16:35	
2-Nitrophenol	ug/L	ND	10.0	4.2	10/28/22 16:35	
3,3'-Dichlorobenzidine	ug/L	ND	20.0	3.1	10/28/22 16:35	
4,6-Dinitro-2-methylphenol	ug/L	ND	50.0	8.2	10/28/22 16:35	
4-Bromophenylphenyl ether	ug/L	ND	10.0	2.9	10/28/22 16:35	
4-Chloro-3-methylphenol	ug/L	ND	20.0	3.0	10/28/22 16:35	
4-Chlorophenylphenyl ether	ug/L	ND	10.0	2.7	10/28/22 16:35	
4-Nitrophenol	ug/L	ND	50.0	5.1	10/28/22 16:35	
Acenaphthene	ug/L	ND	10.0	1.8	10/28/22 16:35	
Acenaphthylene	ug/L	ND	10.0	1.9	10/28/22 16:35	
Anthracene	ug/L	ND	10.0	1.9	10/28/22 16:35	
Benzidine	ug/L	ND	50.0	6.0	10/28/22 16:35	
Benzo(a)anthracene	ug/L	ND	10.0	1.7	10/28/22 16:35	
Benzo(a)pyrene	ug/L	ND	10.0	2.3	10/28/22 16:35	
Benzo(b)fluoranthene	ug/L	ND	10.0	2.6	10/28/22 16:35	
Benzo(g,h,i)perylene	ug/L	ND	10.0	2.2	10/28/22 16:35	
Benzo(k)fluoranthene	ug/L	ND	10.0	2.6	10/28/22 16:35	
bis(2-Chloroethoxy)methane	ug/L	ND	10.0	3.1	10/28/22 16:35	
bis(2-Chloroethyl) ether	ug/L	ND	10.0	2.2	10/28/22 16:35	
bis(2-Chloroisopropyl) ether	ug/L	ND	10.0	3.6	10/28/22 16:35	
bis(2-Ethylhexyl)phthalate	ug/L	ND	5.0	4.2	10/28/22 16:35	
Butylbenzylphthalate	ug/L	ND	10.0	3.7	10/28/22 16:35	
Chrysene	ug/L	ND	10.0	1.9	10/28/22 16:35	
Di-n-butylphthalate	ug/L	ND	10.0	3.3	10/28/22 16:35	
Di-n-octylphthalate	ug/L	ND	10.0	7.3	10/28/22 16:35	
Dibenz(a,h)anthracene	ug/L	ND	10.0	2.6	10/28/22 16:35	
Diethylphthalate	ug/L	ND	10.0	3.2	10/28/22 16:35	

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

METHOD BLANK: 3226363 Matrix: Water  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Dimethylphthalate	ug/L	ND	10.0	2.3	10/28/22 16:35	
Fluoranthene	ug/L	ND	10.0	1.8	10/28/22 16:35	
Fluorene	ug/L	ND	10.0	2.0	10/28/22 16:35	
Hexachloro-1,3-butadiene	ug/L	ND	10.0	3.8	10/28/22 16:35	
Hexachlorobenzene	ug/L	ND	10.0	3.3	10/28/22 16:35	
Hexachlorocyclopentadiene	ug/L	ND	20.0	4.5	10/28/22 16:35	N2
Hexachloroethane	ug/L	ND	10.0	3.4	10/28/22 16:35	N2
Indeno(1,2,3-cd)pyrene	ug/L	ND	10.0	2.6	10/28/22 16:35	
Isophorone	ug/L	ND	10.0	1.9	10/28/22 16:35	
N-Nitroso-di-n-propylamine	ug/L	ND	10.0	3.0	10/28/22 16:35	
N-Nitrosodimethylamine	ug/L	ND	20.0	3.5	10/28/22 16:35	
N-Nitrosodiphenylamine	ug/L	ND	10.0	2.2	10/28/22 16:35	
Naphthalene	ug/L	ND	10.0	2.4	10/28/22 16:35	
Nitrobenzene	ug/L	ND	10.0	3.1	10/28/22 16:35	
Pentachlorophenol	ug/L	ND	50.0	6.7	10/28/22 16:35	
Phenanthrene	ug/L	ND	10.0	1.9	10/28/22 16:35	
Phenol	ug/L	ND	10.0	1.2	10/28/22 16:35	
Pyrene	ug/L	ND	10.0	2.0	10/28/22 16:35	
2,4,6-Tribromophenol (S)	%	98	27-125		10/28/22 16:35	
2-Fluorobiphenyl (S)	%	66	32-92		10/28/22 16:35	
2-Fluorophenol (S)	%	51	9-74		10/28/22 16:35	
Nitrobenzene-d5 (S)	%	79	15-314		10/28/22 16:35	
p-Terphenyl-d14 (S)	%	96	8-146		10/28/22 16:35	
Phenol-d5 (S)	%	36	8-424		10/28/22 16:35	

LABORATORY CONTROL SAMPLE: 3226364

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
1,2,4-Trichlorobenzene	ug/L	50	29.5	59	44-142	
1,2-Dichlorobenzene	ug/L	50	26.9	54	31-79	
1,2-Diphenylhydrazine	ug/L	50	44.3	89	59-111	N2
1,3-Dichlorobenzene	ug/L	50	24.6	49	28-73	
1,4-Dichlorobenzene	ug/L	50	25.0	50	29-76	
2,4,6-Trichlorophenol	ug/L	50	49.2	98	37-144	
2,4-Dichlorophenol	ug/L	50	49.1	98	39-135	
2,4-Dimethylphenol	ug/L	50	47.1	94	32-120	
2,4-Dinitrophenol	ug/L	50	44.2J	88	1-191	
2,4-Dinitrotoluene	ug/L	50	49.4	99	39-139	
2,6-Dinitrotoluene	ug/L	50	49.7	99	50-158	
2-Chloronaphthalene	ug/L	50	40.1	80	60-120	
2-Chlorophenol	ug/L	50	43.8	88	23-134	
2-Nitrophenol	ug/L	50	45.8	92	29-182	
3,3'-Dichlorobenzidine	ug/L	50	48.5	97	1-262	

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

LABORATORY CONTROL SAMPLE: 3226364

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
4,6-Dinitro-2-methylphenol	ug/L	50	44.9J	90	1-181	
4-Bromophenylphenyl ether	ug/L	50	44.9	90	53-127	
4-Chloro-3-methylphenol	ug/L	50	53.9	108	22-147	
4-Chlorophenylphenyl ether	ug/L	50	44.7	89	25-158	
4-Nitrophenol	ug/L	50	23.1J	46	1-132	
Acenaphthene	ug/L	50	43.3	87	47-145	
Acenaphthylene	ug/L	50	44.5	89	33-145	
Anthracene	ug/L	50	46.4	93	27-133	
Benzidine	ug/L	50	7.8J	16	1-64	
Benzo(a)anthracene	ug/L	50	48.4	97	33-143	
Benzo(a)pyrene	ug/L	50	45.5	91	17-163	
Benzo(b)fluoranthene	ug/L	50	48.4	97	24-159	
Benzo(g,h,i)perylene	ug/L	50	44.5	89	1-219	
Benzo(k)fluoranthene	ug/L	50	42.7	85	11-162	
bis(2-Chloroethoxy)methane	ug/L	50	43.0	86	33-184	
bis(2-Chloroethyl) ether	ug/L	50	41.2	82	12-158	
bis(2-Chloroisopropyl) ether	ug/L	50	44.9	90	36-166	
bis(2-Ethylhexyl)phthalate	ug/L	50	47.0	94	8-158	
Butylbenzylphthalate	ug/L	50	50.7	101	1-152	
Chrysene	ug/L	50	47.4	95	17-168	
Di-n-butylphthalate	ug/L	50	46.3	93	1-120	
Di-n-octylphthalate	ug/L	50	45.1	90	4-146	
Dibenz(a,h)anthracene	ug/L	50	43.7	87	1-227	
Diethylphthalate	ug/L	50	46.9	94	1-120	
Dimethylphthalate	ug/L	50	47.8	96	1-120	
Fluoranthene	ug/L	50	48.2	96	26-137	
Fluorene	ug/L	50	47.1	94	59-121	
Hexachloro-1,3-butadiene	ug/L	50	22.6	45	24-120	
Hexachlorobenzene	ug/L	50	44.2	88	1-152	
Hexachlorocyclopentadiene	ug/L	50	20.8	42	5-92 N2	
Hexachloroethane	ug/L	50	20.9	42	40-120 N2	
Indeno(1,2,3-cd)pyrene	ug/L	50	43.7	87	1-171	
Isophorone	ug/L	50	44.2	88	21-196	
N-Nitroso-di-n-propylamine	ug/L	50	46.1	92	1-230	
N-Nitrosodimethylamine	ug/L	50	22.8	46	1-107	
N-Nitrosodiphenylamine	ug/L	50	46.3	93	65-108	
Naphthalene	ug/L	50	34.6	69	21-133	
Nitrobenzene	ug/L	50	41.7	83	35-180	
Pentachlorophenol	ug/L	50	38.9J	78	14-176	
Phenanthrene	ug/L	50	46.9	94	54-120	
Phenol	ug/L	50	19.6	39	5-120	
Pyrene	ug/L	50	49.4	99	52-120	
2,4,6-Tribromophenol (S)	%			97	27-125	
2-Fluorobiphenyl (S)	%			74	32-92	
2-Fluorophenol (S)	%			51	9-74	
Nitrobenzene-d5 (S)	%			82	15-314	
p-Terphenyl-d14 (S)	%			91	8-146	

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**REPORT OF LABORATORY ANALYSIS**

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

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LABORATORY CONTROL SAMPLE: 3226364

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Phenol-d5 (S)	%.			34	8-424	

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**REPORT OF LABORATORY ANALYSIS**

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**QUALITY CONTROL DATA**

Project: Thermal Bioassay Study  
Pace Project No.: 50328663

QC Batch: 701708 Analysis Method: EPA 335.4  
QC Batch Method: EPA 335.4 Analysis Description: 335.4 Cyanide, Total  
Laboratory: Pace Analytical Services - Indianapolis  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

METHOD BLANK: 3225730 Matrix: Water  
Associated Lab Samples: 50328663001, 50328663002, 50328663003, 50328663004, 50328663005, 50328663006, 50328663007, 50328663008

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Cyanide	mg/L	ND	0.0050	0.0018	10/20/22 14:59	

LABORATORY CONTROL SAMPLE: 3225731

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Cyanide	mg/L	0.1	0.095	95	90-110	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 3225732 3225733

Parameter	Units	50328682005 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Cyanide	mg/L	ND	0.1	0.1	0.091	0.098	90	96	90-110	7	20	

MATRIX SPIKE SAMPLE: 3225734

Parameter	Units	50328778001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Cyanide	mg/L	ND	0.1	0.096	92	90-110	

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## QUALIFIERS

Project: Thermal Bioassay Study

Pace Project No.: 50328663

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### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

TNTC - Too Numerous To Count

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit - The lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Reported results are not rounded until the final step prior to reporting. Therefore, calculated parameters that are typically reported as "Total" may vary slightly from the sum of the reported component parameters.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

### ANALYTE QUALIFIERS

H7 Re-extraction or re-analysis could not be performed within method holding time.

L2 Analyte recovery in the laboratory control sample (LCS) was below QC limits. Results for this analyte in associated samples may be biased low.

N2 The lab does not hold NELAC/TNI accreditation for this parameter but other accreditations/certifications may apply. A complete list of accreditations/certifications is available upon request.

S0 Surrogate recovery outside laboratory control limits.

## REPORT OF LABORATORY ANALYSIS

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**QUALITY CONTROL DATA CROSS REFERENCE TABLE**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
50328663001	20-CON-10172022	EPA 608.3	701642	EPA 608.3	701882
50328663002	20-UPS-10172022	EPA 608.3	701642	EPA 608.3	701882
50328663003	20-EFF-10172022	EPA 608.3	701642	EPA 608.3	701882
50328663004	20-DNS-10172022	EPA 608.3	701642	EPA 608.3	701882
50328663005	30-CON-10172022	EPA 608.3	701642	EPA 608.3	701882
50328663006	30-UPS-10172022	EPA 608.3	701642	EPA 608.3	701882
50328663007	30-EFF-10172022	EPA 608.3	701642	EPA 608.3	701882
50328663008	30-DNS-10172022	EPA 608.3	701642	EPA 608.3	701882
50328663001	20-CON-10172022	EPA 608.3	701644	EPA 608.3	701883
50328663002	20-UPS-10172022	EPA 608.3	701644	EPA 608.3	701883
50328663003	20-EFF-10172022	EPA 608.3	701644	EPA 608.3	701883
50328663004	20-DNS-10172022	EPA 608.3	701644	EPA 608.3	701883
50328663005	30-CON-10172022	EPA 608.3	701644	EPA 608.3	701883
50328663006	30-UPS-10172022	EPA 608.3	701644	EPA 608.3	701883
50328663007	30-EFF-10172022	EPA 608.3	701644	EPA 608.3	701883
50328663008	30-DNS-10172022	EPA 608.3	701644	EPA 608.3	701883
50328663001	20-CON-10172022	EPA 200.8	701458	EPA 200.8	701629
50328663002	20-UPS-10172022	EPA 200.8	701458	EPA 200.8	701629
50328663003	20-EFF-10172022	EPA 200.8	701458	EPA 200.8	701629
50328663004	20-DNS-10172022	EPA 200.8	701458	EPA 200.8	701629
50328663005	30-CON-10172022	EPA 200.8	701458	EPA 200.8	701629
50328663006	30-UPS-10172022	EPA 200.8	701458	EPA 200.8	701629
50328663007	30-EFF-10172022	EPA 200.8	701458	EPA 200.8	701629
50328663008	30-DNS-10172022	EPA 200.8	701458	EPA 200.8	701629
50328663001	20-CON-10172022	EPA 245.1	702185	EPA 245.1	702230
50328663002	20-UPS-10172022	EPA 245.1	702185	EPA 245.1	702230
50328663003	20-EFF-10172022	EPA 245.1	702185	EPA 245.1	702230
50328663004	20-DNS-10172022	EPA 245.1	702185	EPA 245.1	702230
50328663005	30-CON-10172022	EPA 245.1	702185	EPA 245.1	702230
50328663006	30-UPS-10172022	EPA 245.1	702185	EPA 245.1	702230
50328663007	30-EFF-10172022	EPA 245.1	702185	EPA 245.1	702230
50328663008	30-DNS-10172022	EPA 245.1	702185	EPA 245.1	702230
50328663001	20-CON-10172022	EPA 625.1	701847	EPA 625.1	703071
50328663002	20-UPS-10172022	EPA 625.1	701847	EPA 625.1	703071
50328663003	20-EFF-10172022	EPA 625.1	701847	EPA 625.1	703071
50328663004	20-DNS-10172022	EPA 625.1	701847	EPA 625.1	703071
50328663005	30-CON-10172022	EPA 625.1	701847	EPA 625.1	703071
50328663006	30-UPS-10172022	EPA 625.1	701847	EPA 625.1	703071
50328663007	30-EFF-10172022	EPA 625.1	701847	EPA 625.1	703071
50328663008	30-DNS-10172022	EPA 625.1	701847	EPA 625.1	703071
50328663001	20-CON-10172022	EPA 624.1	701585	EPA 624.1	701585
50328663002	20-UPS-10172022	EPA 624.1	701585	EPA 624.1	701585
50328663003	20-EFF-10172022	EPA 624.1	701585	EPA 624.1	701585
50328663004	20-DNS-10172022	EPA 624.1	701585	EPA 624.1	701585
50328663005	30-CON-10172022	EPA 624.1	701585	EPA 624.1	701585
50328663006	30-UPS-10172022	EPA 624.1	701585	EPA 624.1	701585

**REPORT OF LABORATORY ANALYSIS**

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**QUALITY CONTROL DATA CROSS REFERENCE TABLE**

Project: Thermal Bioassay Study

Pace Project No.: 50328663

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
50328663007	30-EFF-10172022	EPA 624.1	701585		
50328663008	30-DNS-10172022	EPA 624.1	701585		
50328663001	20-CON-10172022	EPA 335.4	701708	EPA 335.4	701845
50328663002	20-UPS-10172022	EPA 335.4	701708	EPA 335.4	701845
50328663003	20-EFF-10172022	EPA 335.4	701708	EPA 335.4	701845
50328663004	20-DNS-10172022	EPA 335.4	701708	EPA 335.4	701845
50328663005	30-CON-10172022	EPA 335.4	701708	EPA 335.4	701845
50328663006	30-UPS-10172022	EPA 335.4	701708	EPA 335.4	701845
50328663007	30-EFF-10172022	EPA 335.4	701708	EPA 335.4	701845
50328663008	30-DNS-10172022	EPA 335.4	701708	EPA 335.4	701845

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**SAMPLE CONDITION UPON RECEIPT FORM**

Date/Time and Initials of person examining contents: 10/18/22 1330 JAT

1. Courier:  FED EX  UPS  CLIENT  PACE  USPS  OTHER Just
2. Custody Seal on Cooler/Box Present:  Yes  No  
 (If yes) Seals Intact:  Yes  No (leave blank if no seals were present)
3. Thermometer: 1 2 3 4 5 6 A B C D E F (C)
4. Cooler Temperature(s): 1.4/1.4 1.5/1.5 1.8/1.8 2.2/1.9  
 (Initial/Corrected) RECORD TEMPS OF ALL COOLERS RECEIVED (use Comments below to add more)

5. Packing Material:  Bubble Wrap  Bubble Bags  
 None  Other \_\_\_\_\_
6. Ice Type:  Wet  Blue  None
7. If temp. is over 6°C or under 0°C, was the PM notified?:  Yes  No  
 Cooler temp should be above freezing to 6°C

All discrepancies will be written out in the comments section below.

	Yes	No		Yes	No	N/A
USDA Regulated Soils? (HI, ID, NY, WA, OR, CA, NM, TX, OK, AR, LA, TN, AL, MS, NC, SC, GA, FL, or Puerto Rico)		<input checked="" type="checkbox"/>	All containers needing acid/base preservation have been pH CHECKED?: Exceptions: VOA, coliform, LLHg, O&G, RAD CHEM, and any container with a septum cap or preserved with HCl.	<input checked="" type="checkbox"/>		
Short Hold Time Analysis (48 hours or less)? Analysis: <u>2 day</u>	<input checked="" type="checkbox"/>		Circle: HNO3 (<2) <u>H2SO4 (&lt;2)</u> <u>NaOH (&gt;10)</u> NaOH/ZnAc (>9) Any non-conformance to pH recommendations will be noted on the container count form	<input checked="" type="checkbox"/>		
Time 5035A TC placed in Freezer or Short Holds To Lab Time: <u>1515</u>			Residual Chlorine Check (SVOC 625 Pest/PCB 608)	Present	Absent <input checked="" type="checkbox"/>	N/A
Rush TAT Requested (4 days or less):	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Residual Chlorine Check (Total/Amenable/Free Cyanide)		<input checked="" type="checkbox"/>	
Custody Signatures Present?	<input checked="" type="checkbox"/>		Headspace Wisconsin Sulfide?			<input checked="" type="checkbox"/>
Containers Intact?:	<input checked="" type="checkbox"/>		Headspace in VOA Vials (>6mm): See Containter Count form for details	Present	Absent	No VOA Vials Sent <input checked="" type="checkbox"/>
Sample Label (IDs/Dates/Times) Match COC?: Except TCs, which only require sample ID	<input checked="" type="checkbox"/>		Trip Blank Present?		<input checked="" type="checkbox"/>	
Extra labels on Terracore Vials? (soils only)		<input checked="" type="checkbox"/>	Trip Blank Custody Seals?:			<input checked="" type="checkbox"/>

COMMENTS: Rest of temperatures; C = 1.7/1.4, 1.4/1.1, 2.0/1.7, F: 2.2/2.2.





Authored By: Carla Frye	<b><u>Marathon Petroleum Company LP</u></b> <b>Refining</b> Characterization of Sample (Checklist)	Doc No.: 122.21 Rev No: 4
Doc Custodian: Supply Chain Assistant		Illinois Refining Division Supply Chain Procedure
Approved By: Supply Chain Manager	Next Review Date: 12/7/19	Effective Date: 12/7/18
Date Approved: 11/30/15		

**CHARACTERIZATION OF SAMPLE (checklist)**

- SEPARATE FORM REQUIRED FOR EACH SAMPLE THAT DIFFERS IN COMPOSITION. MULTIPLE SAMPLES HAVING THE SAME COMPOSITION MAY BE LISTED ON ONE CHECKLIST.
- THIS CHECKLIST MUST BE ATTACHED TO REFINERY SAMPLE SHIPPING INSTRUCTION FORM. WRITE SAMPLE NAME(S), BELOW, TO MATCH NAME ON SHIPPING INSTRUCTIONS:

**SAMPLE(S):** Robinson Creek Water (Upstream & Downstream of Outfall 001)

If sample has unknown components, or is not well characterized, please take it to a Lab Chemist for characterization, and for help completing this form.

**Check All That Apply**

- Water** Identify source or type: Robinson Creek
- Bio-mass (WWTP bugs/tank microbes/PTP sample)**
- Caustic** pH = \_\_\_\_\_ Get Lab pH analysis, if not known.
- Acid** pH = \_\_\_\_\_ Get Lab pH analysis, if not known.
- Crude Oil**
- Gasoline or other naphtha(s)** Identify source: \_\_\_\_\_
- Diesel / other middle distillates** Identify source: \_\_\_\_\_
- Kerosene**
- Jet Fuel**
- Gas Oil**      AGO      LVGO      HVGO      LCCO
- Slurry / Clarified Oil / #6 Fuel / Bunker Fuel**
- Reduced Crude / Vacuum Column Bottoms**
- Petroleum Coke, including coke fines**
- Sour Water**
- H<sub>2</sub>S** If sample contains H<sub>2</sub>S, check this box   
(Includes rich sponge oil, rich amine, sour waters, sour feeds and sour products)
- Amine** If sample is primarily amine, identify source: \_\_\_\_\_  
And check:      Rich      Lean
- Catalyst** Identify source: \_\_\_\_\_  
And check:      Fresh      Spent      Regenerated
- Elemental Sulfur**
- Heavy Metals** (zinc, mercury, lead or any other metals in more than trace amounts)
- Iron Sulfide / corrosion by-products** Identify source: \_\_\_\_\_
- LPG or other compressed gas** Type: \_\_\_\_\_
- Other:** \_\_\_\_\_

Authored By: <u>Carla Frye</u> Doc Custodian: Supply Chain Assistant Approved By: Supply Chain Manager Date Approved: 12/9/16	<b><u>Marathon Petroleum Company LP</u></b> <b>Refining</b>  <b>Refinery Sample Shipping Instruction Form</b>	Doc No.: 122.20 Rev No: 7  <b>Illinois Refining Division</b> <b>Supply Chain Procedure</b>  Next Review Date: 3/29/20 Effective Date: 3/29/19
----------------------------------------------------------------------------------------------------------------------------------------	------------------------------------------------------------------------------------------------------------------------	--------------------------------------------------------------------------------------------------------------------------------------------------------------------

## REFINERY SAMPLE SHIPPING INSTRUCTION FORM

From: **MARATHON PETROLEUM COMPANY LP**  
**REFINERY WEST GATE**  
**ATTN: WAREHOUSE**  
**ROBINSON IL 62454**

Date: 8/17/22

- SEPARATE FORM REQUIRED FOR EACH SAMPLE.
- SDS SHEETS FOR EACH SAMPLE MUST BE INCLUDED WITH THIS FORM.
- IF FORM NOT COMPLETED, SAMPLE WILL BE RETURNED TO SENDER FOR FURTHER ANALYSIS, RESULTING IN DELAYED DELIVERY TIMES TO SHIP TO LOCATION.

**Proper SDS Name:** MPC Waste Water, Refinery

**SDS Provided by:**  **Sample Provider**     **Robinson Lab**

**Must Arrive At Destination By:**   Same day  

**Container Type:** Glass/plastic                      **Samples on Ice**  **Yes**     **No**

**Flash Point:** Not Available                      \*\*                      **Signature:** \_\_\_\_\_

**Initial Boiling Point:** 100C / 212F                      \*\*                      **Signature:** \_\_\_\_\_

**Quantity:** 2 coolers

**Sample Description:** Robinson Creek Water (Upstream & Downstream of Outfall 001)

\*\* if info provided by other than SDS, signature required

**SHIP TO ADDRESS:**  
 (to be filled in by requestor)

**WAREHOUSE USE ONLY**

Pace Analytical Services  
7726 Moller Road  
Indianapolis, IN 46268

DATE RECEIVED \_\_\_\_\_

WEIGHT IN LBS \_\_\_\_\_

DATE SHIPPED \_\_\_\_\_

VIA \_\_\_\_\_

BY \_\_\_\_\_

**ATTN:** Kenneth Hunt

Hazardous                     

**PHONE:** (317) 228-3120

Non-Hazardous



# SAFETY DATA SHEET

SDS ID NO.: 0317MAR020  
Revision Date 05/27/2015

## 1. IDENTIFICATION

**Product Name:** MPC Waste Water, Refinery  
**Synonym:** Refinery Waste Water; Waste Water Refinery  
**Product Code:** 0317MAR020  
**Chemical Family:** No information available  
**Recommended Use:** Refinery Stream.  
**Restrictions on Use:** All others.

**Manufacturer, Importer, or Responsible Party Name and Address:**  
**MARATHON PETROLEUM COMPANY LP**  
539 South Main Street  
Findlay, OH 45840

**SDS information:** 1-419-421-3070  
**Emergency Telephone:** 1-877-627-5463

## 2. HAZARD IDENTIFICATION

### Classification

#### **OSHA Regulatory Status**

This chemical is not considered hazardous by the 2012 OSHA Hazard Communication Standard (29 CFR 1910.1200)

#### **Hazards Not Otherwise Classified (HNOC)**

Not applicable.

### Label elements

#### **EMERGENCY OVERVIEW**

No known significant effects or critical hazards.

<b>Appearance</b> Clear or Colored Liquid	<b>Physical State</b> Liquid	<b>Odor</b> Slight Hydrocarbon
-------------------------------------------	------------------------------	--------------------------------

**Precautionary Statements - Prevention**  
Not applicable.

**Precautionary Statements - Response**  
Not applicable.

**Precautionary Statements - Storage**

Sensitivity to Mechanical Impact No.  
Sensitivity to Static Discharge No.

**Special protective equipment and precautions for firefighters**

Firefighters should wear full protective clothing and positive-pressure self-contained breathing apparatus (SCBA) with a full face-piece, as appropriate. Avoid using straight water streams. Use water spray to cool exposed surfaces from as far a distance as possible.

**Additional firefighting tactics**

Not applicable.

**NFPA** Health 1 Flammability 1 Instability 0 Special Hazard -

**6. ACCIDENTAL RELEASE MEASURES**

- Personal precautions:** Keep public away. Isolate and evacuate area. Shut off source if safe to do so.
- Protective equipment:** Use personal protection measures as recommended in Section 8.
- Emergency procedures:** Advise authorities and National Response Center (800-424-8802) if the product has entered a water course or sewer. Notify local health and pollution control agencies, if appropriate.
- Environmental precautions:** Avoid release to the environment. Avoid subsoil penetration.
- Methods and materials for containment:** Contain liquid with sand or soil.
- Methods and materials for cleaning up:** Use suitable absorbent materials such as vermiculite, sand, or clay to clean up residual liquids. Recover and return free product to proper containers.

**7. HANDLING AND STORAGE**

- Safe Handling Precautions:** Avoid repeated and prolonged skin contact. Avoid breathing fumes, gas, or vapors. Use only with adequate ventilation. Use personal protection measures as recommended in Section 8. Do not expose to heat, open flames, strong oxidizers or other sources of ignition. Exercise good personal hygiene including removal of soiled clothing and prompt washing with soap and water. Refer to applicable EPA, OSHA, NFPA and consistent state and local requirements.
- Storage Conditions:** Store in properly closed containers that are appropriately labeled and in a cool, well-ventilated area.
- Incompatible Materials** Strong oxidizing agents.

**8. EXPOSURE CONTROLS/PERSONAL PROTECTION**

Name	ACGIH TLV	OSHA PELS:	OSHA - Vacated PELs	NIOSH IDLH
Dissolved Hydrocarbons Mixture	-	-	-	-

**Notes:** The manufacturer has voluntarily elected to provide exposure limits contained in OSHA's 1989 air contaminants standard in its SDSs, even though certain of those exposure limits were vacated in 1992.

**Engineering measures:** Local or general exhaust required when using at elevated temperatures that generate vapors or mists.

**Personal protective equipment**

**Incompatible Materials**

Strong oxidizing agents.

**Hazardous decomposition products**

None known under normal conditions of use.

**11. TOXICOLOGICAL INFORMATION**

**Potential short-term adverse effects from overexposures**

- Inhalation** Prolonged excessive exposure may cause irritation to the respiratory tract.
- Eye contact** Exposure to vapor or contact with liquid may cause mild eye irritation, including tearing, stinging, and redness.
- Skin contact** Prolonged and repeated contact may cause defatting and drying of the skin and may lead to irritation and/or dermatitis.
- Ingestion** Ingestion of large amounts may cause gastrointestinal disturbances.

**Acute toxicological data**

Name	Oral LD50	Dermal LD50	Inhalation LC50
Dissolved Hydrocarbons Mixture	-	-	-

**Delayed and immediate effects as well as chronic effects from short and long-term exposure**

This product may contain small amounts of aromatic hydrocarbons (benzene, toluene, xylene and ethyl benzene). These materials are not present in sufficient quantities to produce an acutely toxic response. This product may also contain small amounts of heavy metals (lead, chromium, arsenic) that are not present in sufficient quantities to produce an acutely toxic response.

**Adverse effects related to the physical, chemical and toxicological characteristics**

- Signs and Symptoms** Repeated or prolonged skin contact may cause drying, reddening, itching and cracking.
- Sensitization** Not expected to be a skin or respiratory sensitizer.
- Mutagenic effects** None known.

**Carcinogenicity** Cancer designations are listed in the table below

Name	ACGIH (Class)	IARC (Class)	NTP	OSHA
Dissolved Hydrocarbons Mixture	Not Listed	Not Listed	Not Listed	Not Listed

- Reproductive toxicity** None known.
- Specific Target Organ Toxicity (STOT) - single exposure** Not classified.
- Specific Target Organ Toxicity (STOT) - repeated exposure** Not classified.
- Aspiration hazard** No data available.

**12. ECOLOGICAL INFORMATION**

**Ecotoxicity** This product is not expected to be harmful to aquatic organisms.

Name	Algae/aquatic plants	Fish	Toxicity to	Crustacea

Hazardous Substance (EHS) List:

Name	CERCLA/SARA - Section 302 Extremely Hazardous Substances and TPQs
Dissolved Hydrocarbons	NA

**SARA Section 304:** This product may contain component(s) identified either as an EHS or a CERCLA Hazardous substance which in case of a spill or release may be subject to SARA reporting requirements:

Name	Hazardous Substances RQs
Dissolved Hydrocarbons	NA

**SARA Section 311/312:** The following EPA hazard categories apply to this product:  
None

**SARA Section 313:** This product may contain component(s), which if in exceedance of the de minimus threshold, may be subject to the reporting requirements of SARA Title III Section 313 Toxic Release Reporting (Form R).

Name	CERCLA/SARA 313 Emission reporting:
Dissolved Hydrocarbons	None

**State and Community Right-To-Know Regulations:**

The following component(s) of this material are identified on the regulatory lists below:

- Dissolved Hydrocarbons
  - Louisiana Right-To-Know: Not Listed
  - California Proposition 65: Not Listed
  - New Jersey Right-To-Know: Not Listed
  - Pennsylvania Right-To-Know: Not Listed
  - Massachusetts Right-To-Know: Not Listed
  - Florida Substance List: Not Listed
  - Rhode Island Right-To-Know: Not Listed
  - Michigan Critical Materials Register List: Not Listed
  - Massachusetts Extraordinarily Hazardous Substances: Not Listed
  - California - Regulated Carcinogens: Not Listed
  - Pennsylvania RTK - Special Hazardous Substances: Not Listed
  - New Jersey - Special Hazardous Substances: Not Listed
  - New Jersey - Environmental Hazardous Substances List: Not Listed
  - Illinois - Toxic Air Contaminants: Not Listed
  - New York - Reporting of Releases Part 597 - List of Hazardous Substances: Not Listed

**Canada DSL/NDL Inventory:** This product and/or its components are listed either on the Domestic Substances List (DSL) or are exempt.

**Canadian Regulatory Information:** This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations and the SDS contains all of the information required by those regulations.

**Note:** Not applicable.

**16. OTHER INFORMATION**

**Prepared By** Toxicology and Product Safety

Revision Notes



www.pacelabs.com

**Report Prepared for:**

Kenneth Hunt  
PACE Indianapolis  
7726 Moller Road  
Indianapolis IN 46268

**REPORT OF  
LABORATORY  
ANALYSIS FOR  
TCDD**

**Report Information:**

**PaceProject#: 10630428**  
**Sample Receipt Date: 10/20/2022**  
**Client Project #: 50328663**  
**Client Sub PO #: N/A**  
**State Cert #: N/A**

**Invoicing & Reporting Options:**

The report provided has been invoiced as a Level 2 2,3,7,8-TCDD Report. If an upgrade of this report package is requested, an additional charge may be applied.

Please review the attached invoice for accuracy and forward any questions to Carolynne Trout, your Pace Project Manager.

**This report has been reviewed by:**

October 27, 2022

Carolynne Trout, Project Manager  
(612) 607-6351  
(612) 607-6444 (fax)  
Carolynne.Trout@pacelabs.com



**Report of Laboratory Analysis**

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The results relate only to the samples included in this report.

**Report Prepared Date:**

October 27, 2022





## **DISCUSSION**

This report presents the results from the analyses performed on eight samples submitted by a representative of Pace Analytical Services, LLC. The samples were analyzed for the presence or absence of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) using USEPA Method 1613B. The reporting limits were set to correspond to the lowest calibration point and a nominal 1-Liter sample amount, and the sensitivity was verified by signal-to-noise measurements. The quantitation limits, adjusted for sample extraction amount, may be somewhat higher or lower than the reporting limits provided in this report.

The recoveries of the isotopically-labeled TCDD internal standard in the sample extracts ranged from 50-57%. All of the labeled standard recoveries obtained for this project were within the target ranges specified in Method 1613B. Also, since the quantification of the native TCDD was based on isotope dilution, the data were automatically corrected for recovery and accurate values were obtained.

A laboratory method blank was prepared and analyzed with the sample batch as part of our routine quality control procedures. The results show the blank to be free of 2,3,7,8-TCDD at the reporting limit.

Laboratory spike samples were also prepared using clean reference matrix that had been fortified with native standard material. The results show that the spiked native TCDD was recovered at 115-119% with a relative percent difference of 3.4%. These results were within the target ranges for the method. Matrix spikes were not prepared with the sample batch.

## **REPORT OF LABORATORY ANALYSIS**

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## Minnesota Laboratory Certifications

Authority	Certificate #	Authority	Certificate #
A2LA	2926.01	Mississippi	MN00064
Alabama	40770	Missouri	10100
Alaska-DW	MN00064	Montana	CERT0092
Alaska-UST	17-009	Nebraska	NE-OS-18-06
Arizona	AZ0014	Nevada	MN00064
Arkansas - WW	88-0680	New Hampshire	2081
Arkansas-DW	MN00064	New Jersey	MN002
California	2929	New York	11647
Colorado	MN00064	North Carolina-	27700
Connecticut	PH-0256	North Carolina-	530
Florida	E87605	North Dakota	R-036
Georgia	959	Ohio-DW	41244
Hawaii	MN00064	Ohio-VAP (170	CL101
Idaho	MN00064	Ohio-VAP (180	CL110
Illinois	200011	Oklahoma	9507
Indiana	C-MN-01	Oregon- rimary	MN300001
Iowa	368	Oregon-Second	MN200001
Kansas	E-10167	Pennsylvania	68-00563
Kentucky-DW	90062	Puerto Rico	MN00064
Kentucky-WW	90062	South Carolina	74003
Louisiana-DEQ	AI-84596	Tennessee	TN02818
Louisiana-DW	MN00064	Texas	T104704192
Maine	MN00064	Utah	MN00064
Maryland	322	Vermont	VT-027053137
Michigan	9909	Virginia	460163
Minnesota	027-053-137	Washington	C486
Minnesota-Ag	via MN 027-053	West Virginia-D	382
Minnesota-Petr	1240	West Virginia-D	9952C
		Wisconsin	999407970
		Wyoming-UST	via A2LA 2926.

## REPORT OF LABORATORY ANALYSIS

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Report No.....10630428

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**Pace Analytical Services, LLC**  
1700 Elm Street, Suite 200  
Minneapolis, MN 55414  
Phone: 612.607.1700  
Fax: 612.607.6444  
www.pacelabs.com

## **Appendix A**

### **Sample Management**

## **REPORT OF LABORATORY ANALYSIS**

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# Internal Transfer Chain of Custody



Samples Pre-Logged into eCOC.

State Of Origin: IL  Yes  No  
 Cert. Needed:  Yes  No

Workorder: 50328663 Workorder Name: Thermal Bioassay Study

Owner Received Date: 10/18/2022 Results Requested By: 11/1/2022

Report To  
 Kenneth Hunt  
 Pace Analytical Indianapolis  
 7726 Moller Road  
 Indianapolis, IN 46268  
 Phone (317)228-3100

Subcontract To  
 Pace Analytical Minnesota  
 1700 Elm Street  
 Suite 200  
 Minneapolis, MN 55414  
 Phone (612)607-1700



WO#: 10630428



Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	Preserved Containers		LAB USE ONLY
						Unpreserved		
1	20-CON-10172022	PS	10/17/2022 10:55	50328663001	Water	2		001
2	20-UPS-10172022	PS	10/17/2022 10:40	50328663002	Water	2		002
3	20-EFF-10172022	PS	10/17/2022 10:20	50328663003	Water	2		003
4	20-DNS-10172022	PS	10/17/2022 09:50	50328663004	Water	2		004
5	30-CON-10172022	PS	10/17/2022 12:45	50328663005	Water	2		005
6	30-UPS-10172022	PS	10/17/2022 12:25	50328663006	Water	2		006
7	30-EFF-10172022	PS	10/17/2022 12:05	50328663007	Water	2		007
8	30-DNS-10172022	PS	10/17/2022 11:35	50328663008	Water	2		008

Method 1613  
 Dioxin to Pace MN  
 03,7,8-TCDD only

Transfers	Released By	Date/Time	Received By	Date/Time
1	<i>[Signature]</i>	10-19-22 15:39	FedEx	
2	<i>[Signature]</i>		Mary Pace	10/20/22 9:50
3				


2,3,7,8-TCDD only via 1613

Cooler Temperature on Receipt 0.8/0.9°C

Received on Ice  or  N

Samples Intact  or  N

\*\*\*In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document. This chain of custody is considered complete as is since this information is available in the owner laboratory.

	<b>DC#_Title: ENV-FRM-MIN4-0142 v02_Sample Condition Upon Receipt (SCUR) Exception Form</b>
	<b>Effective Date: 09/22/2022</b>

Workorder #: \_\_\_\_\_

No Temp Blank		
Read Temp	Corrected Temp	Average temp

<b>PM Notified of Out of Temp Cooler?</b> <input type="checkbox"/> Yes <input type="checkbox"/> No If yes, indicate who was contacted, date and time. If no, indicate reason why. _____
<b>Multiple Cooler Project?</b> <input type="checkbox"/> Yes <input type="checkbox"/> No

If anything is OVER 6.0° C, you **MUST** document containers in this section **HERE**



Tracking Number	Temperature
6075 7334 8871	0.8
6075 7334 8882	0.9

Out of Temp Sample ID	Container Type	# of Containers

pH Adjustment Log for Preserved Samples										
Sample ID	Type Of Preserve	pH Upon Receipt	Date Adjusted	Time Adjusted	Amount Added (mL)	Lot # Added	pH After	In Compliance After Addition?		Initials
								<input type="checkbox"/> Yes	<input type="checkbox"/> No	
								<input type="checkbox"/> Yes	<input type="checkbox"/> No	
								<input type="checkbox"/> Yes	<input type="checkbox"/> No	
								<input type="checkbox"/> Yes	<input type="checkbox"/> No	
								<input type="checkbox"/> Yes	<input type="checkbox"/> No	
								<input type="checkbox"/> Yes	<input type="checkbox"/> No	
								<input type="checkbox"/> Yes	<input type="checkbox"/> No	
								<input type="checkbox"/> Yes	<input type="checkbox"/> No	

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Effective Date:

Sample Condition Upon Receipt Client Name: Pace Analytical Indianapolis Project #: **WO# : 10630428**

Courier:  FedEx  UPS  USPS  Client  
 Pace  SpeedDee  Commercial

PM: CT1 Due Date: 10/27/22  
 CLIENT: PASI-INDI

See Exceptions ENV-FRM-MIN4-0142

Tracking Number: \_\_\_\_\_

Custody Seal on Cooler/Box Present?  Yes  No Seals Intact?  Yes  No Biological Tissue Frozen?  Yes  No  N/A

Packing Material:  Bubble Wrap  Bubble Bags  None  Other Temp Blank?  Yes  No

Thermometer:  T1 (0461)  T2 (1336)  T3 (0459)  T4 (0254)  T5 (0178)  
 T6 (0235)  T7 (0042)  T8 (0775)  01339252/1710

Type of Ice:  Wet  Blue  Dry  None  
 Melted

Did Samples Originate In West Virginia?  Yes  No Were All Container Temps Taken?  Yes  No  N/A

Temp should be above freezing to 6 °C Cooler temp Read w/Temp Blank: \_\_\_\_\_ °C Average Corrected Temp (no temp blank only): \_\_\_\_\_ °C

Correction Factor: \_\_\_\_\_ Cooler Temp Corrected w/temp blank: \_\_\_\_\_ °C  See Exceptions ENV-FRM-MIN4-0142  1 Container

USDA Regulated Soil:  N/A  water sample  other: \_\_\_\_\_ Date/Initials of Person Examining Contents: 10/10/22 NV

Did samples originate in a quarantine zone within the United States: AL, AR, AZ CA, FL, GA, ID, LA, MS, NC, NM, NY, OK, OR, SC, TN, TX, or VA (check maps)?  Yes  No

Did samples originate from a foreign source (Internationally, including Hawaii and Puerto Rico)?  Yes  No

If Yes to either question, fill out a Regulated Soil Checklist (ENV-FRM-MIN4-0154) and include with SCUR/COC paperwork.

Location (Check one):	Duluth	<input checked="" type="checkbox"/> Minneapolis	Virginia	COMMENTS
Chain of Custody Present and Filled Out?	<input type="checkbox"/> Yes	<input checked="" type="checkbox"/> No	<input type="checkbox"/> N/A	1.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes	<input type="checkbox"/> No	<input type="checkbox"/> N/A	2.
Sampler Name and/or Signature on COC?	<input type="checkbox"/> Yes	<input checked="" type="checkbox"/> No	<input type="checkbox"/> N/A	3.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes	<input type="checkbox"/> No	<input type="checkbox"/> N/A	4. If fecal: <input type="checkbox"/> <8 hrs <input type="checkbox"/> >8 hr, <24 <input type="checkbox"/> No
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/> Yes	<input checked="" type="checkbox"/> No	<input type="checkbox"/> N/A	5. <input type="checkbox"/> Facal Collform <input type="checkbox"/> HPC <input type="checkbox"/> Total Collform/E.coli <input type="checkbox"/> BOD/cBOD <input type="checkbox"/> Hex Chrom <input type="checkbox"/> Turbidity <input type="checkbox"/> Nitrate <input type="checkbox"/> Nitrite <input type="checkbox"/> Orthophos <input type="checkbox"/> Other
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes	<input checked="" type="checkbox"/> No	<input type="checkbox"/> N/A	6.
Sufficient Sample Volume?	<input checked="" type="checkbox"/> Yes	<input type="checkbox"/> No	<input type="checkbox"/> N/A	7.
Correct Containers Used?	<input checked="" type="checkbox"/> Yes	<input type="checkbox"/> No	<input type="checkbox"/> N/A	8.
-Pace Containers Used?	<input checked="" type="checkbox"/> Yes	<input type="checkbox"/> No	<input type="checkbox"/> N/A	9.
Containers Intact?	<input checked="" type="checkbox"/> Yes	<input type="checkbox"/> No	<input type="checkbox"/> N/A	10. Is sediment visible in the dissolved container? <input type="checkbox"/> Yes <input type="checkbox"/> No
Field Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	11. If no, write ID/Date/Time of container below: <input type="checkbox"/> See Exceptions ENV-FRM-MIN4-0142
Is sufficient information available to reconcile the samples to the COC? Matrix: <input checked="" type="checkbox"/> Water <input type="checkbox"/> Soil <input type="checkbox"/> Oil <input type="checkbox"/> Other	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	12. Sample # <input type="checkbox"/> NaOH <input type="checkbox"/> HNO3 <input type="checkbox"/> H2SO4 <input type="checkbox"/> Zinc Acetate Positive for Residual Chlorine? <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> See Exceptions ENV-FRM-MIN4-0142 pH Paper Lot # Residual Chlorine <input type="checkbox"/> 0-6 Roll <input type="checkbox"/> 0-6 Strip <input type="checkbox"/> 0-14 Strip
All containers needing acid/base preservation have been checked?	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	13.
All containers needing preservation are found to be in compliance with EPA recommendation? (HNO3, H2SO4, <2pH, NaOH >9 Sulfide, NaOH >10 Cyanide)	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	14.
Exceptions: VOA, Collform, TOC/DOC Oil and Grease, DRO/8015 (water) and Dioxins/PFAS (*If adding preservative to a container, it must be added to associated field and equipment blanks--verify with PM first.)	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	15.
Headspace In Methyl Mercury Container?	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	
Extra labels present on soil VOA or WIDRO containers?	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	<input type="checkbox"/> See Exceptions ENV-FRM-MIN4-0142
Headspace In VOA Vials (greater than 6mm)?	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	
3 Trip Blanks Present?	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes	<input type="checkbox"/> No	<input checked="" type="checkbox"/> N/A	Pace Trip Blank Lot # (if purchased): _____

CLIENT NOTIFICATION/RESOLUTION Field Data Required?  Yes  No

Person Contacted: \_\_\_\_\_ Date/Time: \_\_\_\_\_

Comments/Resolution: \_\_\_\_\_

Project Manager Review: Matt Ray Date: 10/21/22

NOTE: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e., out of hold, incorrect preservative, out of temp, incorrect containers).

Labeled By: NV Line: (2)



## Reporting Flags

- A = Reporting Limit based on signal to noise (EDL)
- B = Less than 10x higher than method blank level
- C = Result obtained from confirmation analysis
- D = Result obtained from analysis of diluted sample
- E = Exceeds calibration range
- I = Isotope ratio out of specification
- J = Estimated value
- L = Suppressive interference, analyte may be biased low
- Nn = Value obtained from additional analysis
- P = PCDE Interference
- R = Recovery outside target range
- S = Peak saturated
- U = Analyte not detected
- V = Result verified by confirmation analysis
- X = %D Exceeds limits
- Y = Calculated using average of daily RFs
- \* = See Discussion

## REPORT OF LABORATORY ANALYSIS

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## **Appendix B**

### **Sample Analysis Summary**

## **REPORT OF LABORATORY ANALYSIS**

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	20-CON-10172022		
Lab Sample ID	50328663001		
Filename	L221026A_03		
Injected By	SMT		
Total Amount Extracted	980 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	10/17/2022 10:55
ICAL ID	L220811	Received	10/20/2022 08:50
CCal Filename(s)	L221026A_01	Extracted	10/21/2022 10:15
Method Blank ID	BLANK-102002	Analyzed	10/26/2022 10:26

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	52
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	59

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	20-UPS-10172022		
Lab Sample ID	50328663002		
Filename	L221026A_04		
Injected By	SMT		
Total Amount Extracted	960 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	10/17/2022 10:40
ICAL ID	L220811	Received	10/20/2022 08:50
CCal Filename(s)	L221026A_01	Extracted	10/21/2022 10:15
Method Blank ID	BLANK-102002	Analyzed	10/26/2022 11:09

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	50
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	55

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

**REPORT OF LABORATORY ANALYSIS**

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	20-EFF-10172022		
Lab Sample ID	50328663003		
Filename	L221026A_05		
Injected By	SMT		
Total Amount Extracted	992 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	10/17/2022 10:20
ICAL ID	L220811	Received	10/20/2022 08:50
CCal Filename(s)	L221026A_01	Extracted	10/21/2022 10:15
Method Blank ID	BLANK-102002	Analyzed	10/26/2022 11:52

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	56
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	61

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

**REPORT OF LABORATORY ANALYSIS**

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	20-DNS-10172022		
Lab Sample ID	50328663004		
Filename	L221025B_02		
Injected By	SMT		
Total Amount Extracted	993 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	10/17/2022 09:50
ICAL ID	L220811	Received	10/20/2022 08:50
CCal Filename(s)	L221025A_18	Extracted	10/21/2022 10:15
Method Blank ID	BLANK-102002	Analyzed	10/26/2022 00:15

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	57
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	60

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

**REPORT OF LABORATORY ANALYSIS**

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	30-CON-10172022		
Lab Sample ID	50328663005		
Filename	L221025B_03		
Injected By	SMT		
Total Amount Extracted	980 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	10/17/2022 12:45
ICAL ID	L220811	Received	10/20/2022 08:50
CCal Filename(s)	L221025A_18	Extracted	10/21/2022 10:15
Method Blank ID	BLANK-102002	Analyzed	10/26/2022 00:58

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	56
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	63

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	30-UPS-10172022		
Lab Sample ID	50328663006		
Filename	L221025B_04		
Injected By	SMT		
Total Amount Extracted	983 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	10/17/2022 12:25
ICAL ID	L220811	Received	10/20/2022 08:50
CCal Filename(s)	L221025A_18	Extracted	10/21/2022 10:15
Method Blank ID	BLANK-102002	Analyzed	10/26/2022 01:41

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	57
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	62

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

**REPORT OF LABORATORY ANALYSIS**

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**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	30-EFF-10172022		
Lab Sample ID	50328663007		
Filename	L221025B_05		
Injected By	SMT		
Total Amount Extracted	990 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	10/17/2022 12:05
ICAL ID	L220811	Received	10/20/2022 08:50
CCal Filename(s)	L221025A_18	Extracted	10/21/2022 10:15
Method Blank ID	BLANK-102002	Analyzed	10/26/2022 02:24

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	51
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	55

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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 Fax: 612-607-6444

**Method 1613B Sample Analysis Results**

Client - PACE Indianapolis

Client's Sample ID	30-DNS-10172022		
Lab Sample ID	50328663008		
Filename	L221025B_06		
Injected By	SMT		
Total Amount Extracted	991 mL	Matrix	Water
% Moisture	NA	Dilution	NA
Dry Weight Extracted	NA	Collected	10/17/2022 11:35
ICAL ID	L220811	Received	10/20/2022 08:50
CCal Filename(s)	L221025A_18	Extracted	10/21/2022 10:15
Method Blank ID	BLANK-102002	Analyzed	10/26/2022 03:07

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	---	10	2,3,7,8-TCDD-13C	2.00	56
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	63

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

ND = Not Detected  
 NA = Not Applicable  
 NC = Not Calculated

R = Recovery outside target range  
 E = Exceeds calibration range

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**Method 1613B Blank Analysis Results**

Lab Sample Name	DFBLKQD	Matrix	Water
Lab Sample ID	BLANK-102002	Dilution	NA
Filename	U221025A_14	Extracted	10/21/2022 10:15
Total Amount Extracted	1020 mL	Analyzed	10/25/2022 11:50
ICAL ID	U221005	Injected By	SMT
CCal Filename(s)	U221024A_18		

Native Isomers	Conc pg/L	EMPC pg/L	RL pg/L	Internal Standards	ng's Added	Percent Recovery
2,3,7,8-TCDD	ND	—	10	2,3,7,8-TCDD-13C	2.00	56
				Recovery Standard 1,2,3,4-TCDD-13C	2.00	NA
				Cleanup Standard 2,3,7,8-TCDD-37Cl4	0.20	66

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).  
 EMPC = Estimated Maximum Possible Concentration  
 RL = Reporting Limit

**REPORT OF LABORATORY ANALYSIS**

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**Method 1613B Laboratory Control Spike Results**

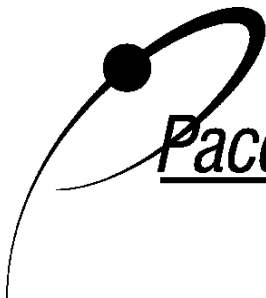
Lab Sample ID	LCS-102003	Matrix	Water
Filename	U221025A_11	Dilution	NA
Total Amount Extracted	1030 mL	Extracted	10/21/2022 10:15
ICAL ID	U221005	Analyzed	10/25/2022 09:29
CCal Filename	U221024A_18	Injected By	SMT
Method Blank ID	BLANK-102002		

Compound	Cs	Cr	Lower Limit	Upper Limit	% Rec.
2,3,7,8-TCDD	10	12	7.3	14.6	119
2,3,7,8-TCDD-37Cl4	10	7.8	3.7	15.8	78
2,3,7,8-TCDD-13C	100	54	25.0	141.0	54

Cs = Concentration Spiked (ng/mL)  
 Cr = Concentration Recovered (ng/mL)  
 Rec. = Recovery (Expressed as Percent)  
 Control Limit Reference: Method 1613, Table 6, 10/94 Revision  
 R = Recovery outside of control limits  
 Nn = Value obtained from additional analysis  
 \* = See Discussion

**REPORT OF LABORATORY ANALYSIS**

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**Method 1613B Laboratory Control Spike Results**

Lab Sample ID	LCSD-102004	Matrix	Water
Filename	U221025A_12	Dilution	NA
Total Amount Extracted	1030 mL	Extracted	10/21/2022 10:15
ICAL ID	U221005	Analyzed	10/25/2022 10:15
CCal Filename	U221024A_18	Injected By	SMT
Method Blank ID	BLANK-102002		

Compound	Cs	Cr	Lower Limit	Upper Limit	% Rec.
2,3,7,8-TCDD	10	11	7.3	14.6	115
2,3,7,8-TCDD-37Cl4	10	8.3	3.7	15.8	83
2,3,7,8-TCDD-13C	100	65	25.0	141.0	65

Cs = Concentration Spiked (ng/mL)  
 Cr = Concentration Recovered (ng/mL)  
 Rec. = Recovery (Expressed as Percent)  
 Control Limit Reference: Method 1613, Table 6, 10/94 Revision  
 R = Recovery outside of control limits  
 Nn = Value obtained from additional analysis  
 \* = See Discussion

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**Method 1613B**

**Spike Recovery Relative Percent Difference (RPD) Results**

Client PACE Indianapolis

Spike 1 ID LCS-102003  
Spike 1 Filename U221025A\_11

Spike 2 ID LCSD-102004  
Spike 2 Filename U221025A\_12

<b>Compound</b>	<b>Spike 1 %REC</b>	<b>Spike 2 %REC</b>	<b>%RPD</b>
2,3,7,8-TCDD	119	115	3.4

%REC = Percent Recovered

RPD = The difference between the two values divided by the mean value

**REPORT OF LABORATORY ANALYSIS**

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**CERTIFICATE OF SERVICE**

I, Melissa S. Brown, the undersigned, on oath state the following:

That I have served the attached **MARATHON PETROLEUM COMPANY LP'S**  
**SUBMITTAL OF REPORT**, via electronic mail upon:

Don Brown  
Clerk of the Board  
Illinois Pollution Control Board  
100 W. Randolph Street, Suite 11-500  
Chicago, Illinois 60601  
Don.Brown@illinois.gov

Carol Webb  
Hearing Officer  
Illinois Pollution Control Board  
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That my email address is Melissa.Brown@heplerbroom.com.

That the number of pages in the email transmission is 542 pages.

That the email transmission took place before 5:00 p.m. on the date of October 5, 2023.

/s/ Melissa S. Brown

Melissa. S. Brown

Date: October 5, 2023