

Matagorda Bay Mitigation Trust 2022-2023 Funding Cycle
RFP # 2022-2023-1

**Title: Reproductive & Developmental Toxicity of “Forever Chemicals” to
Matagorda Bay’s prey fishes Reproductive & Developmental Toxicity of “Forever
Chemicals” to Matagorda Bay’s prey fishes**

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Q1 October 2023 Progress Report

Q1 Update:

We have nearly completed the sampling needed to characterize the PFAS profile of Matagorda Bay as part of Phase 1. To date, we have currently completed environmental sampling at three of our four proposed sampling locations (see Figure 1). Samples are in the process of being prepared for PFAS analysis.

Sample Collection:

We have collected samples from three of our four sampling locations. Samples were collected from the reference site (Figure 2), downstream of the effluent from the Palacios Wastewater Treatment Plant (Figure 3), and downstream from the effluent of both the Point Comfort Wastewater Treatment Plant and the Formosa Plastics Corporation (Figure 4). At each location, samples were taken in triplicate from 3 sites along a 20 meter transect (Figures 2-4). At each sampling site along the transects, a total of three water samples were collected in PFAS-free bottles. Following water collection, five 5cm sediment cores were taken at the same location and placed in a PFAS-free bottle. In addition to sample collection, each site had the following recorded: latitude + longitude, salinity, dissolved oxygen, pH, and temperature.

In total, 27 water samples and 9 composite sediment samples were collected at each site. Following collection, all samples were immediately placed on ice and transported to the University of Texas at Austin Marine Science Institute (UT MSI). Samples were placed at -20°C until processing for analysis. Storage at -20°C is the most accepted pre-analysis storage, and has been shown to keep PFAS stable for more >6 months prior to analysis¹.

Sample Analyses:

Sediment samples (9 per site) are in the process of being sent to SGS AXYS Analytical Services, LTD for PFAS Analysis. These samples will be extracted and analyzed following EPA Draft Method 1633 “821-D-21-001: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS” (Figure 5), a method that identifies 40 PFAS (Table 1). SGS AXYS Analytical Services, LTD have received accreditation for EPA Method 1633 from the US Department of Defense. This analysis will provide a full profile of the 40 most prevalent PFAS (Table 1) and will provide the necessary data to create a PFAS profile for Matagorda Bay. These data will also provide PFAS profiles specific to each

point source identified. Samples will also be assessed for dissolved organic carbon (DOC), as DOC is known to significantly influence bioavailability of PFAS.

Water samples (27 per site) are currently being prepared for analysis following EPA Method 531.1 “*Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid*” (Figure 5), a method that will be performed at UT MSI. This method will the presence of 22 PFAS (Table 2). Water samples are currently undergoing PFAS extraction using Solid Phase Extraction (SPE) following Method 531.1 (Figure 5). Briefly, each water sample is defrosted from -20°C, spiked with the EPA 537.1 Method isotopically-labelled internal standards, run through the PFAS-free resin SPE columns (Bond Elut-LMS, 500mg 6mL; Agilent Technologies) recommended by the EPA, and placed at -20°C until elution. Samples will be sent to the Analytical Core Laboratory at UT MSI once eluted for PFAS analysis on the IM Q-TOF LC-MS.

To date, the Analytical Core Laboratory has developed the LC and QTOF methods following EPA Method 531.1, confirmed PFAS are retained, and confirmed they can be correctly identified. The Analytical Core Laboratory has also determined detection limits and are performing the necessary standard curve calibrations. Following this calibration process, SPE columns will be eluted and samples will be transferred to the Analytical Core Laboratory for analysis. Samples will also be assessed for dissolved organic carbon (DOC), as DOC is known to significantly influence bioavailability of PFAS. These analyses will provide the necessary data to characterize the PFAS present in water samples taken from each sampling location in Matagorda Bay.

Preparation for Phases 2 and 3:

In addition to sample collection at each sampling site, we have also identified locations for seining for collection of sheepshead minnows at each site (Figures 2-4). Sheepshead minnows will be collected for analysis of PFAS body burdens using EPA Method 1633 from these locations for Phase 2. These same sites will be used to collect sheepshead minnows for transport to UT MSI for the chronic PFAS exposures and reproductive / fecundity studies detailed in Phase 3.

References:

¹Woudneh, Million B., Bharat Chandramouli, Coreen Hamilton, and Richard Grace. "Effect of sample storage on the quantitative determination of 29 PFAS: observation of analyte interconversions during storage." *Environmental Science & Technology* 53, no. 21 (2019): 12576-12585.

²<https://www.sgsaxys.com/2021/09/14/epa-announces-availability-of-epa-1633-draft-pfas-method-developed-by-sgs-axys-sgs-axys-continues-to-expand-range-of-pfas-testing-methods/>

Figures:



Figure 1. Expected point sources for introduction of PFAS into the Matagorda Bay system and the proposed sampling sites for characterization of PFAS in the Bay.

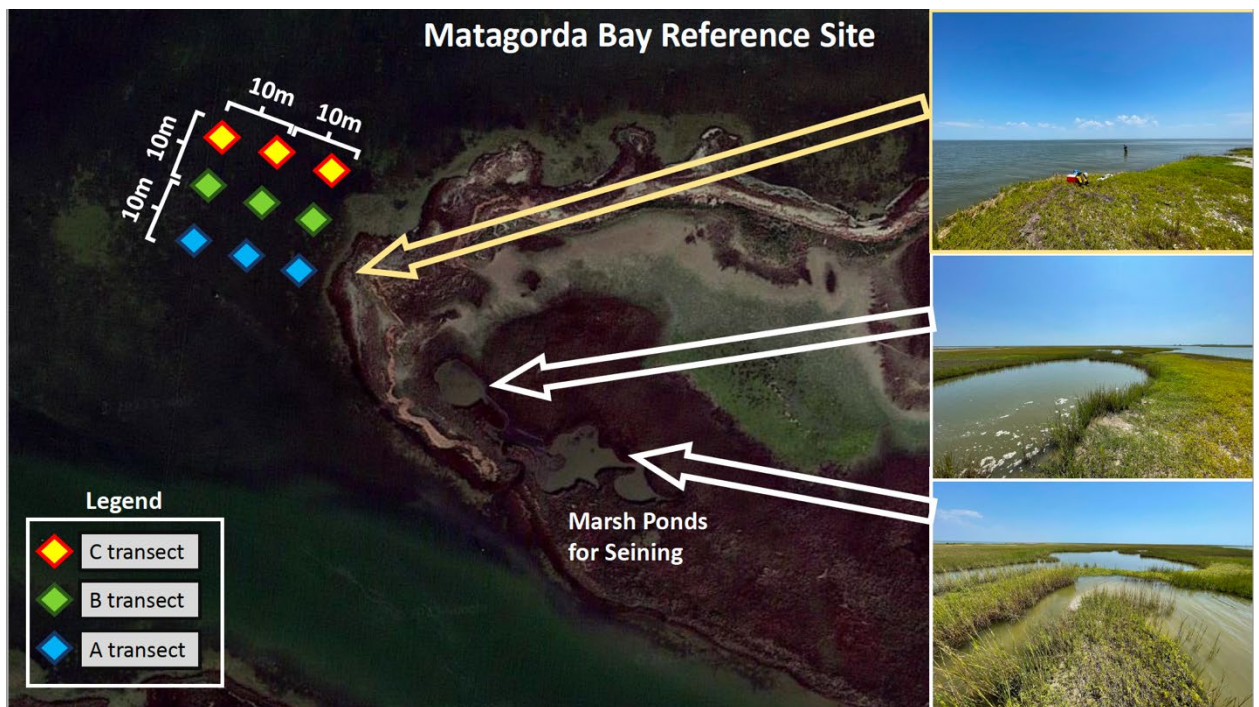


Figure 2. Site sampled as the 'Reference Site' for PFAS characterization of Matagorda Bay.

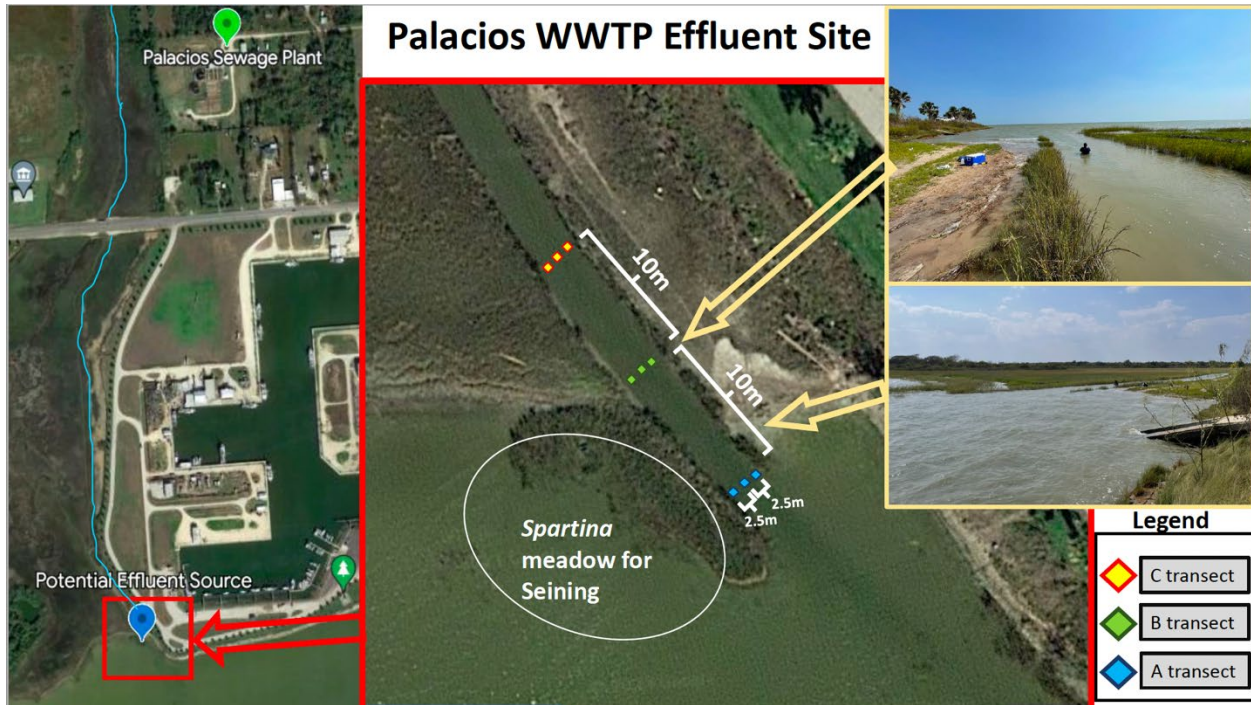


Figure 3. Site sampled as an expected point source of PFAS for PFAS characterization of Matagorda Bay near the Palacios Wastewater Treatment Plant.

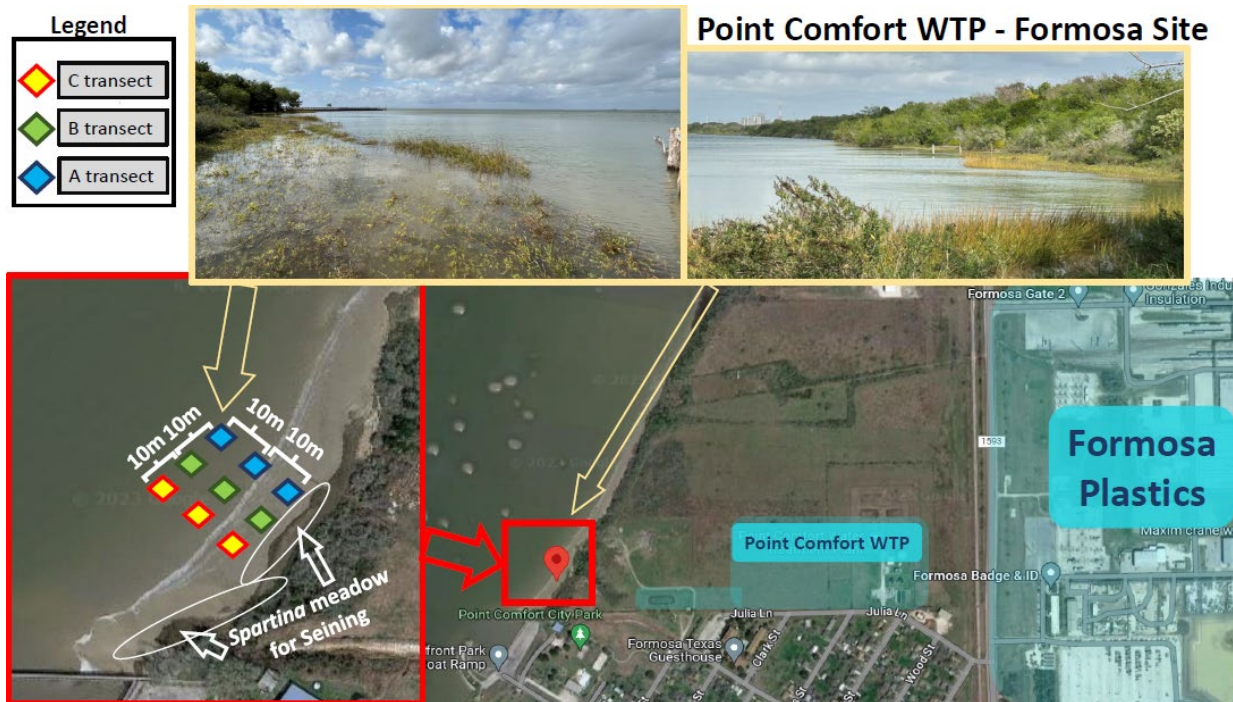


Figure 4. Site sampled as an expected point source of PFAS for PFAS characterization of Matagorda Bay near Comfort Point Wastewater Treatment Plant and Formosa Plastics Corporation.

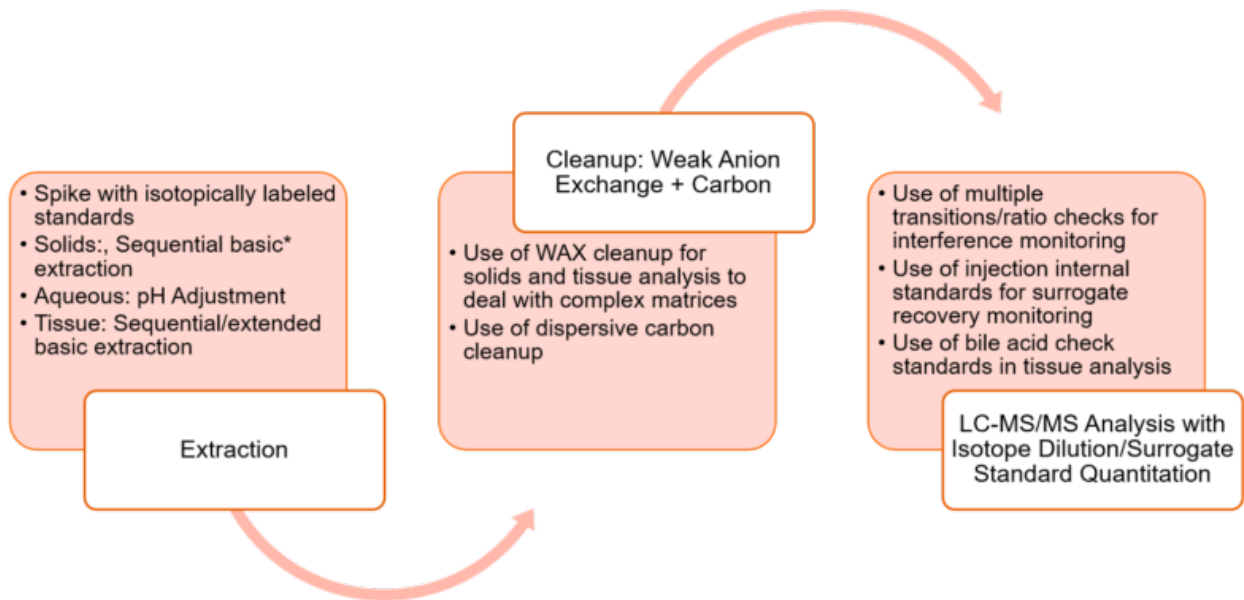


Figure 5. Extraction and analysis methods used for water and sediment samples for PFAS analysis using EPA Methods 1633 and 537.1. Schematic provided by SGS AXYS Analytical Services, LTD².

Tables:

FAMILY	ANALYTE	TYPICAL REPORTING LIMITS
PFCA	PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnA, PFDoA, PFTrDA, PFTetraDA	<input type="checkbox"/> 0.4-1.6 ng/L water <input type="checkbox"/> 0.04-0.16 ng/g solid <input type="checkbox"/> 0.1-0.4 ng/g tissue <input type="checkbox"/> 0.1-0.4 ng/mL serum <input type="checkbox"/> 10-40 ppb AFFF
PFSA	PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS, PFDoS	<input type="checkbox"/> 0.4 ng/L water <input type="checkbox"/> 0.04 ng/g solid <input type="checkbox"/> 0.1 ng/g tissue <input type="checkbox"/> 0.1 ng/mL serum <input type="checkbox"/> 10 ppb AFFF
FTS and FTCA	4:2, 6:2 and 8:2 FTS, 3:3, 5:3 and 7:3 FTCA	<input type="checkbox"/> 3.2- 10 ng/L water <input type="checkbox"/> 0.32 – 1 ng/g solid <input type="checkbox"/> 0.8 – 2.5 ng/g tissue <input type="checkbox"/> 40-250 ppb AFFF
Sulfonamides	EtFOSAA, MeFOSAA, PFOSA, EtFOSA, MeFOSA, EtFOSE and MeFOSE	<input type="checkbox"/> 0.4-4 ng/L water <input type="checkbox"/> 0.04-0.4 ng/g solid <input type="checkbox"/> 0.1-1 ng/g tissue <input type="checkbox"/> 10-100 ppb AFFF
Ether carboxylates and sulfonates	HFPO-DA (GEN-X), ADONA, F-53B, NFDHA, PFMBA, PFMPA, PFEESA	<input type="checkbox"/> 0.4-1.6 ng/L water <input type="checkbox"/> 0.04 – 0.16 ng/g solid <input type="checkbox"/> 0.1-0.4 ng/g tissue <input type="checkbox"/> 10-40 ppb AFFF

Table 1. PFAS to be analyzed using EPA Method 1633 for sediment (i.e., solid) samples taken from each sampling site in Matagorda Bay. Table provided by SGS AXYS Analytical Services, LTD².

Analyte	Internal Standard Reference
PFBS	2
PFHxA	1
HFPO-DA	1
PFHpA	1
PFHxS	2
ADONA	1
PFOA	1
PFOS	2
PFNA	1
9Cl-PF3ONS	2
PFDA	1
NMeFOSAA	3
PFUnA	1
NEtFOSAA	3
11Cl-PF3OUdS	2
PFDoA	1
PFTrDA	1
PFTA	1
¹³ C2-PFHxA	1
¹³ C3-HFPO-DA	1
¹³ C2-PFDA	1
d5-NEtFOSAA	3
¹³ C2-PFOA – IS#1	-
¹³ C4-PFOS – IS#2	-
d3-NMeFOSAA – IS#3	-

Table 2. PFAS to be analyzed using EPA Method 537.1 for water samples taken from each sampling site in Matagorda Bay.