

Matagorda Bay Mitigation Trust 2023-2024 Funding Cycle  
Title: Evaluating Ecological and Human Health Risk of PFAS in Matagorda Bay  
Contract #067  
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Y1 Q3-4; Y2 Q1 2025 Progress Report

**Y1 Q3-4; Y2 Q1 Update:**

We are currently testing water samples taken from each oyster reef in Matagorda Bay for 13 PFAS contamination using our recently optimized methods. We are also processing oyster tissue samples for body burden analyses. We are also currently preparing our facilities for an oyster culture in the Nielsen Lab for Phases 2 and 3.

**Phase 1:**

Water samples collected concurrently with oysters have been prepped for analysis for PFAS contamination using EPA Method 531.1 “*Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid*” to test for the presence of 13 PFAS (Table 1). Prior to solid phase extraction (SPE), all water samples were passed through a 0.7-micron glass micro fibre Whatman filters to remove suspended particulate matter. Samples are filtered to determine the PFAS that are dissolved in water samples and not partitioned to the suspended particulate matter. Following filtering, samples were passed through polystyrenedivinylbenzene (SDVB) containing cartridges (Agilent: Catalog No.1225-5021) to extract method analytes, which were then eluted from the solid phase sorbent with methanol (ThermoFisher; Catalog No. 325740010). After being concentrated to dryness, extract was adjusted to a 90:10% (vol/vol) water/methanol in 100uL. Analysis of PFAS was performed at the Mass Spectrometry Facility of the Department of Chemistry at the University of Texas at Austin in Austin, Texas using a Shimadzu 8060 LC MS/MS Triple Quadrupole. Briefly, 10uL of sample (and standards) were injected and a Purospher Star RP-18 endcapped (3micron) Hibar TR 50-4 delay column was utilized to delay elution, preventing interference during separation by the analytical column. Analytes were then separated using a Waters Crop Acquity UPLC BEH C18 (2.1x50mm, 1.7micron) column. A mobile phase LC gradient consisted of Bottle A [acetonitrile] and Bottle B [ammonium acetate (2mM) in 95:5 water/acetonitrile] beginning at 2%-A and 98%-B, reaching 95%-A and 5%-B at 10 minutes, and ending at 2%-A and 98%-B over 12 minutes. An in house-PFAS standard mix containing 13 reagent grade PFAS stocks was used to generate calibration curves for quantification. Post-analysis data processing (identification and quantification) was performed using LabSolutions Insight software.

We are currently waiting to receive these data to provide profiles of PFAS at each of the oyster reefs sampled within the Matagorda Bay system. These data will be important as we continue the development of Phases 2 and 3, as they will confirm which PFAS we expose oysters to for in-lab experiments and to ensure we do so at environmentally relevant concentrations.

In addition, the method to process and analyze the oyster tissue (i.e., meat) collected is currently underway. We have begun to work through optimization of US EPA method EPA 712-C-16-004 “*Ecological Effects Test Guidelines OCSPP 850.170 Oyster Bioaccumulation Factor (BCF)*”, EPA method 1633, and additional methods published in peer reviewed literature to optimize the extraction protocol for for biotic tissue samples. All the shucked oyster tissue has been divided into ~2-gram aliquots for analyses. We are actively working to optimize these protocols and have performed a variety of preparation methods to determine which tissue preparation method (i.e., freeze drying versus slightly frozen homogenates) results in the highest percent recovery from each tissue. We will continue to modify the protocol to improve our percent recovery of PFAS from oyster tissue and will be able to run samples on both the IM Q-TOF LC-MS and Shimadzu 8060 LC MS/MS Triple Quadrupole. Once our method is optimized and has a sufficient percent recovery, we will be able to analyze each oyster (n=20; 5 per sampling location) for the 13 PFAS shown in Table 1.

**Phases 2 and 3:**

We have hired a Postdoctoral Fellow in the Nielsen lab with previous experience working with oysters in a laboratory setting working to establish an oyster culture in the Nielsen Lab, following best practices via guidance from the experts at the Texas A&M AgriLife Research Mariculture Laboratory in Corpus Christi, TX. We met with Dr. Chris Hollenbeck to discuss logistics for establishing oyster cultures and experimental methods for Phases 2 and 3. We are currently setting up the tank systems for our oyster culture, with our target goal to begin juvenile oyster PFAS exposures in the early fall. These exposures will include environmentally relevant PFAS detected at the oyster reefs within the Matagorda Bay system. We will also work with the Texas A&M AgriLife Research Mariculture Laboratory to coordinate our oyster gamete PFAS exposures with their oyster spawning season to ensure best practices.

<b>Compound</b>	<b>Limit of Detection (ppt)</b>
PFDA	0.3
PFDoA	30
PFTTrDA	30
PFBA	10
PFOA	7.5
PFOS	10
PFHpA	4
PFNA	5
PFHxS	10
PFTeDA	300
PFHxA	17
PFUnA	3

Table 1. PFAS analyzed using a modified EPA Method 537.1 for filtered water samples taken from oyster sampling sites in Matagorda Bay. The limit of detection for each compound is shown here in parts per trillion (ppt).