# EPA/ORD/CESER Quality Assurance Memorandum for dataset from Bunker Hill sediments microcosm study

April 16, 2025

From: Anna Wade

USEPA Office of Research and Development, Center for Environmental Solutions and Emergency Response, Land Remediation and Technology Division

On behalf of coauthors:

Todd Luxton, Matt Noerpel, Jenny Goetz

USEPA Office of Research and Development, Center for Environmental Solutions and Emergency Response, Land Remediation and Technology Division

Mahendra Arambewela

Pegasus Technical Services Inc. (PTSI)

To: Michael Gonzalez, Division Director

USEPA Office of Research and Development, Center for Environmental Solutions and

Emergency Response, Land Remediation and Technology Division

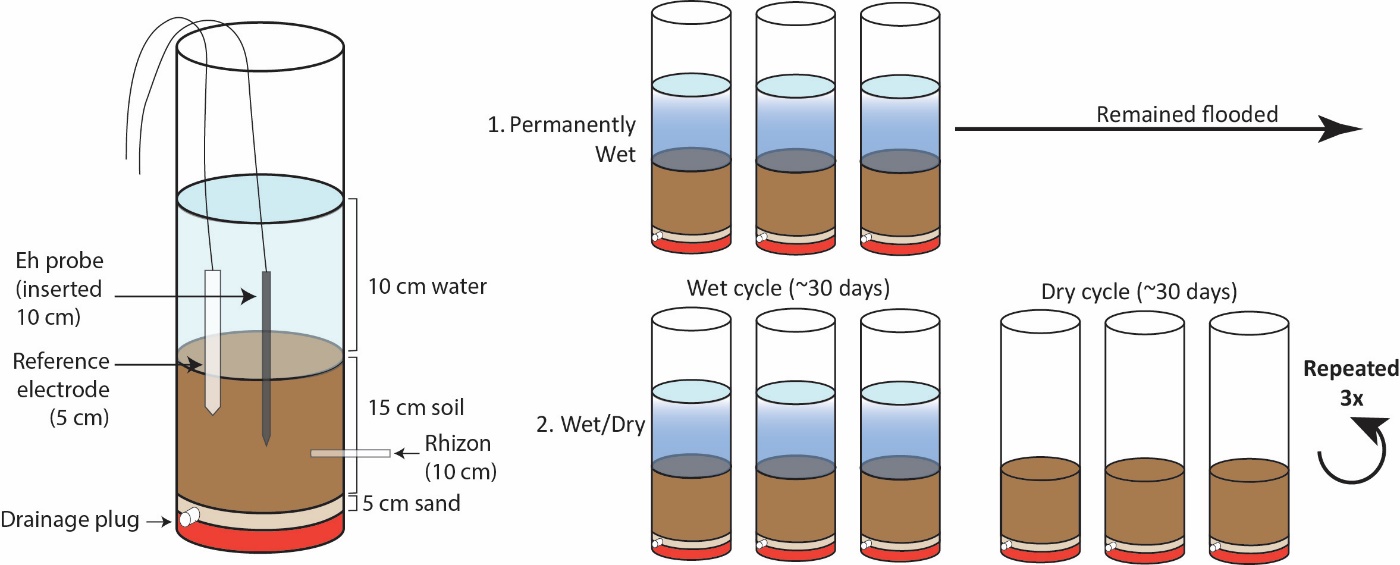
RE: Data from 2023-2024 Bunker Hill microcosm study for publication on EPA ScienceHub

**Summary**

A laboratory-based microcosm study was conducted from September 2023 to July 2024 by the Office of Research (ORD) Cincinnati Lab (ORD-CESER-LRTD-CAISB). QA Category B data was generated following established methodologies and quality-assurance procedures. ORD clearance policy requirements for internal technical review, quality assurance review and supervisor reviewer/approval were met. This data is to be published on ScienceHub following satisfaction of EPA ORD policies. No interpretation of the data is provided. The results of this study will be described in detail in a publication subjected to external, expert peer review.

Sediments, collected from the Lower Coeur d’Alene Basin (CDA) in the Bunker Hill Mining and Metallurgical Complex Superfund Site (Bunker Hill), were used in microcosms set up and monitored by ORD Cincinnati staff. The study was designed to evaluate the impact of repeated wetting and drying cycles on sediment porewater metal concentrations in the Lower CDA. Sediments were subjected to three wetting-drying cycles, and porewater was sampled throughout the duration of the experiment. Two sediment types were subjected to two different treatments (Permanently wet vs wet/dry) with 3 replicates of each, yielding 12 total microcosms. Each wetting-drying cycle lasted 30 days and was followed by 30 days of drying (Figure 1). This data package summarizes sediment, sediment porewater and surface water results including total metal concentrations in sediment and sediment porewater, dissolved organic carbon (DOC) concentrations, dissolved iron and sulfide, UV absorbance at 254 nm, YSI measurements of porewater, sulfate concentrations, times series data of redox potential (Eh), and lead (Pb) speciation analysis of select samples.

This memo details the appropriate QA/QC procedures used for each parameter. All analyses followed standard or established protocols outlined in the metadata. No interpretation or analysis of the data is included.

**Figure 1**. Schematic overview of microcosm set up. Depths in parathesis indicate the depth that the probe, electrode or rhizon was installed at. Microcosms were flooded from the bottom up through the drainage plug and drained by the plug at the end of each cycle (Wet/Dry only).

## 1. Chain of Custody

Sediment samples were received by ORD within three days of the sampling event. Upon receipt, sediment samples were checked for container integrity and proper preservation. All sediment samples were received intact and in good condition. Sediment porewater and surface water samples were preserved following standard QA/QC procedures put forth in standard operating procedures (SOPs) and were disposed of on-site following ORD SHEM requirements.

## 2. Quality Assurance and Control Procedures

The following tables outline the quality assurance and control (QA/QC) procedures implemented by the ORD-CESER-LRTD Cincinnati staff during sample analysis:

| **Analyte, Matrix, and method** | **Quality Control Objective** | **Acceptance Criteria** | | **Description** | **Frequency** | **Method Reference** | **Instrument** | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sample digestion (Solid) | Minimize sample loss during digestion | <5% | | % loss of initial weight following digestion | Each run | K-LRTD-SOP-1193-0  Mars Xrpress | | |
| External NIST standard recovery rate | < +/- 20% | | Analyte recoveries calculated relative to certified values | Each run |
| Method Blank | < MRL | | Process a blank in the exact same manner as samples to ensure no contamination during preparation and analytical process | Each run |
| Total Metals (Liquid and Solid) | Instrument Calibration check | 90-110% | | Percent recovery for analysis of an anion standard during sample analysis | At the beginning of every analysis and once every ten samples | EPA Method 6020B | Agilent 7900 | |
| Instrument duplicate | <10% | | Percent difference of anion concentration for duplicate sample analysis | 10% of samples |
| Matrix spike | 70-130% | | Recovery of added known concentration of analytes. | 10% of samples |
| **pH, specific conductance (SpC), dissolved oxygen (DO), ORP on YSI** | | | | | | | | |
| Dissolved oxygen (DO) | Instrument calibration check | <5% error | | 2-point calibration curve, 0 and 100% | Beginning of analysis | K-LRTD-SOP-1208-0 | YSI Multiparameter Water Quality Sonde | |
| pH | Instrument calibration | Slope > 95% | | 3-point calibration curve | Beginning of analysis |
| Calibration check | Within 0.1 pH units of expected value | | 3-point calibration curve | Beginning of analysis |
| Specific conductance (SpC) | Instrument calibration check | <5% error | | Calibration with 1,000 us/cm standard | Beginning of analysis |
| Oxidation reduction potential (ORP) | Instrument calibration check | <5% error | | Calibration with Zobell solution relative to Ag/AgCl redox couple | Beginning of analysis |
| **Colorimetric measurements** | | | | | | | | |
| Ferrous iron, Fe(II), (Liquid) | Method blank | < 0.1 mg/L or 10% | | < MRL (0.1 mg/L) or <10% lowest sample | Before each sample | HACH-8146 | HACH-1900 |
| Sulfides (Liquid) | Method blank | < 0.1 mg/L or <10% | | < MRL (0.1 mg/L) or <10% of paired sample | Before each sample | K-HSMMD-SOP-2044 | HACH-1900 | |
| **X-Ray Absorption Spectroscopy** | | | | | | | | |
| Speciation (Solid) | Quality control objectives: Fluorescence counts > 1 million, Correct calibration of energy beam. For details on quality control/quality assurance, acceptance criteria and frequency please see Section 5.9 in Method Reference. | | | | | K-LRTD-SOP-1200-0 | Synchrotron | |
| **Redox potential (Eh) time series** | | | | | | | | |
| Redox potential (Eh) | Sensor calibration check | | < 5 mV | Difference between reference electrodes used in two treatments when placed in buffered solution | Beginning of analysis | See footnote1 | SWP/ORP-10-1-C, SWP/REF-12-0-A, SWP/TMP-10-1-C2 | |
| Calibration check | | < 5 mV difference between sensors, < +/- 10 mV from calibration value | Calibration with Tape water, Zobell solution, and Orion ORP relative to Ag/AgCl redox couple | Beginning of analysis |
| Redox potential (Eh) (cont.) | Instrument mainten-ance | | Full | Replenishment of 3M KCl gel | At least every 6 months to 1 year, or upon depletion of solution |
| **Dissolved Organic Carbon / UV-Vis** | | | | | | | | |
| Determination of Non-Purgeable Organic Carbon in Water by Shimadzu TOC-VCPH, High Temperature TOC Analyzer (TOC) | Post calibration accuracy check | 90-110% | | Recovery of secondary source calibration check prior to analyzing samples | Analysis initiation | Method 415.3 | Shimadzu TOC-VCPH analyzer with standard sensitivity catalyst/ Shimadzu 2700 UV/Vis Spectrometer | |
| Accuracy check | 90-110% | | Percent recovery of known concentration of DOC | Every 15 samples or once per analytical batch |  |
| Precision check duplicates | 15% | | Relative percent difference between duplicated sample analyses | 10% of samples or once per analytical batch |  |

1 SWAP Instruments (2024) Manual for SWAP soil Redox probes. Version: 2024-V3. SWAP Instruments B.V., Netherlands, 37 pages. [www.swapinstruments.com](http://www.swapinstruments.com)

2 Sensor model numbers from [www.swapinstruments.com](http://www.swapinstruments.com)

## 3. Data Qualifiers

Data for all samples were assessed for compliance following the QA/QC parameters outlined above. If data did not meet specific requirements set forth by the QAPP then data qualifiers were assigned. In the cases data qualifiers were assigned, the sample was re-analyzed. At this point all QA/QC parameters in the provided data set are in compliance. Elements that were below the detection limit are left blank in the attached spreadsheets.