MEMs Field Deployment

FINAL REPORT
July 2020

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Disclaimer Statement

1. Report No. CAIT-UTC-NC58	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle MEMs Field Deployment		5. Report Date July 2020
		6. Performing Organization Code CAIT/Rutgers University
7. Author(s) Robert Miskewitz Ph.D, Ali Maher Ph.D., Mehdi Javanmard Ph.D., Phillip Sontag Ph.D. and Kelly Francisco Ph.D.		8. Performing Organization Report No. CAIT-UTC-NC58
9. Performing Organization Name and Address Center for Advanced Infrastructure and Transportation Rutgers The State University of New Jersey 100 Brett Road, Piscataway, NJ 08854		10. Work Unit No.
		11. Contract or Grant No. DTRT13-G-UTC28
12. Sponsoring Agency Name and Address Center for Advanced Infrastructure and Transportation Rutgers, The State University of New Jersey 100 Brett Road, Piscataway, NJ 08854		13. Type of Report and Period Covered Final Report July 2018 to December 2019
		14. Sponsoring Agency Code

15. Supplementary Notes
U.S. Department of Transportation/OST-R
1200 New Jersey Avenue, SE
Washington, DC 20590-0001

16. Abstract
Rutgers CAIT personnel recently developed a real-time lead sensor for use in sediment systems. The largest hurdle by far however was the development of a digestion procedure to accurately assess the actual concentration within a given mass of sediment. The sensor that was developed can only measure lead in the dissolved phase, as a result in order to deploy this sensor system a digestion step similar to those used in standard laboratory analyses had to be developed. Standard digestion procedures involve extended periods of time and high doses of acid which could damage our sensor system. As a result, an accelerated enzyme digestion procedure was identified and customized for this application. The procedure was tested and applied to field collected sediment sample and results were compared to standard laboratory procedures (Atomic Adsorption). Results indicate that the accelerated enzyme digestion procedure was successful and gave results that were comparable to the standard laboratory results.

17. Key Words Real-time Sediment Lead Sensor		18. Distribution Statement	
19. Security Classification (of this report) Unclassified	20. Security Classification (of this page) Unclassified	21. No. of Pages 21	22. Price

Form DOT F 1700.7 (8-69)

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1. Purpose and Focus

Recently, researchers at Rutgers have designed, fabricated, and characterized a nano-electronic label-free sensing platform capable of detecting toxic metals in environmental samples. The utility of this platform was demonstrated by developing a sensing system for the detection of lead. The electrochemical sensors were microfabricated using graphene oxide electrodes. Graphene oxide is selectively patterned for use as the working electrode in a 3-electrode measurement. This electrode was characterized using SEM, AFM, and Raman spectroscopy. Square wave stripping voltammetry is used and sensor response is characterized over a dynamic range of four orders of magnitude. The capability to detect lead was demonstrated at concentrations as low as 4 part per billion (ppb) making it suitable for detecting lead in-situ in environmental samples such as ground water and sediment. The sensor was validated using pre-digested samples and laboratory calibration standards. The scope of this project was to take this sensor development a step further in its evolution by developing rapid, accurate and repeatable digestion step that can be integrated into this sensor system.

The standard laboratory procedure for the analysis of lead in aqueous samples involves the use of a strong acid to completely digest all the lead in the sample regardless of the form it is in or the chemical is it is associated with. This approach was deemed to be impractical due to the strength of the acid which would actually digest the sensor along with the lead in the sample. As a result, it was determined that an accelerated enzyme digestion would be the most appropriate approach to dissolving the lead to make it available for the MEMs sensor to detect. In the environment (surface water and sediment water), lead is typically found in specific fractions. Perez-Cid et al. (1999) used a sequential extraction to identify those fractions. They were: the acid soluble fraction which consists of lead bound up in carbonates, sulfates, hydroxide minerals, a reducible fraction which consists of lead bound up by Fe oxides, an oxidizable fraction which consists of lead bound up in organic material and a residual "sulfide" fraction which consists of lead bound up in sulfides (which are often found in organic marsh sediments which is a common target of this sensor). An important aspect of this project was to determine if the accelerated enzyme extraction was successful at digesting each of these lead containing fractions so that sediment lead samples would provide accurate and reproducible results regardless of the sediment characteristics.

The work that was carried out involved several steps and was primarily conducted to develop and verify the applicability of the enzyme digestion procedure for use with the MEMs Pb sensor. The Methods Section of this report provides a step by step description of the materials and methods used complete:

- Sediment Sequential Extraction
- Accelerated Enzyme Extraction
- Inductively coupled plasma optical emission spectroscopy (ICP-OES) of environmental sediment samples
  - Cheesequake Creek, New Jersey (NJ)
- MEMs sensor sediment lead concentrations

The process developed during this study will be used in concert with the results from a related effort to develop field sampling techniques and the appropriate configuration of a sampling device/sensor configuration to field deploy this technology. The method described in the following section is a step by step description of the method used. It is followed by side by side Pb analyses using standard laboratory techniques and the MEMs sensor to develop the model for use with this sensor.

2.0 Materials and Methods

2.1 Sequential Extraction – Sediment Metal Fractions

Sequential extraction procedures are used to locate the occurrence of heavy metals and utilize 3 or 4-step sequential extractions to measure the metal of interest in the exchangeable, carbonate, iron (Fe) and manganese (Mn) oxide, organic (oxidizable), and strong acid-extractable (residual) phases (Okoro et al. 2012). The "Community Bureau of Reference" (BCR) has produced recent sequential extraction methods that use a 3-step extraction method (Elass et al. 2004, Guevara-Riba et al. 2004, Yuan et al. 2004). Traditional sequential extraction protocols (Tessier et al. 1979) and subsequent modifications (Rauret et al. 1989) require slow digestion times and with consistent heating. Accelerated approaches to sequential extraction methods were explored to decrease digestion times, lower equipment and reagent cost, and increase capacity of sediment metal analysis. Microwave heating (Gulmini et al. 1994, Perez-Cid et al. 1999a) and ultrasound (Vaisanen and Kiljunen 2005) techniques have been used to accelerate sequential extraction. Metal yields of ultrasound techniques have been compared to accepted the Tessier protocol (Perez-Cid et al. 1999b, c) with significant differences between metals in the third (Fe and Mn fraction) and fourth (oxidizable) extracts. However, good agreement in metal concentrations were found between ultrasound accelerated and original BCR methods in each of the three chemical fractions (carbonate, Fe and Mn, and oxidizable) (Perez-Cid et al. 1998), including Pb, our toxic metal of interest.

2.2 Sediment Acid Digestion – Residual Calculation

A total metal acid digestion (EPA 1996) was performed to quantify total concentrations of Pb in sediment. These total Pb concentrations were used to estimate the residual phase of Pb in sediments (sulfide bound) requiring a strong acid digestion. The residual phase was calculated through the following equation (Residual Pb = Total Pb – (Fraction 1 (carbonate) + Fraction 2 (Fe and Mn oxides) + Fraction 3 (oxidizable)).

- 50Watt Sonic Dismembrator 110V (Fisher Scientific; Model 50)
- Analytical Balance – accurate weights at 0.001 g
- Centrifuge – 50 mL capacity
- Teflon spatula or spoon
- Freeze dryer (lyophilization)
- 50 mL Falcon centrifuge tubes, polypropylene

- Centrifuge tube racks – 50 mL
- Macropipette and tips (1 – 5mL)
- Glass digestion vessels – 250 mL
- Glass beakers (tops) – 100 mL

2.3 Reagents – Sequential Extraction and Acid Digestion

Organic or inorganic reagents used in digestion shall be of reagent grade (trace metal grade (TMG) or high performance liquid chromatography (HPLC) grade) chemicals and all acid cleaning of digestion surfaces with certified (ACS Plus) hydrochloric acid in dilute concentrations (35% and 10%).

- Acetic acid (concentrated), CH_3COOH.
- Sodium acetate, anhydrous ($\geq$99.995%), CH_3COONa.
- Hydroxylamine hydrochloride ($\geq$99.995%), $NH_2OH \cdot HCl$.
- Hydrogen peroxide (30%), H_2O_2.
- Ammonium acetate (97.0%), $C_2H_7NO_2$.
- 12N Hydrochloric acid (Muriatic acid), $HCl \cdot H_2O$.
- 16 N Nitric acid (67 to 70% w/w), HNO_3.

2.4 Sediment-Enzyme Digest – Procedure

A 0.25 mL of "fresh" (non-lyophilized) sediment sub-sample is drawn from the sample falcon tube (attached to harpoon sampler, filled with "fresh" sediment) into a separate falcon tube (with sponge and filter, occupying about 0.25 mL) used for digestion. Reagents and sediment-enzyme digest parameters are discussed below.

1. Dispense 0.25 mL of sediment from sample falcon tube (no filter) into digest falcon tube (with filter)
2. Add 0.25 mL of beta-glucosidase enzyme (high enzyme activity (1 Kilo Unites (KU)))
3. Mix sample + beta-glucosidase for 7 min
4. Add 0.25 mL of 0.1 M nitric acid
5. Mix sample + beta-glucosidase + 0.1 M nitric acid for 7 min
6. Add 0.50 mL of 0.1 M sodium acetate buffer (needed for sensor measurements)

This protocol yields a total of 1.5 mL (0.25 mL filter + 0.25 mL sample + 0.25 mL enzyme + 0.25 mL acid + 0.5 mL buffer) in the 2.0 mL falcon tube allowing for proper headspace during mixing to increase contact of enzyme and acid with sediment sample.

Table 1 3-step sequential extraction method modified from Perez-Cid et al. 1999 to accommodate smaller weights of sediment samples (~0.5 g).

Fraction	Reagents	Ultrasound Digestion	Metals Removed
F1 – Acid Soluble Fraction	10 mL of Glacial Acetic Acid (0.11 mol L^{-1})	50W – 7 min	Carbonates, sulfates, hydroxide minerals
F2 – Reducible fraction	10 mL Hydroxylamine hydrochloride (0.1 mol L^{-1})	50W – 7 min	Bound to Fe-Mn oxides
F3 – Oxidizable Fraction	5 mL Hydrogen Peroxide (30% w/v)	50W – 2 min	Oxidizable organics, some humic acids
F3 – Oxidizable Fraction (stage 2)	10 mL Ammonium Acetate	50W – 6 min	Oxidizable organics, some humic acids
F4 – Residual "Sulfide" Fraction	**Sulfide [Pb] = Total [Pb] – (F1 [Pb] + F2 [Pb] + F3 [Pb])**		Dissolved and particulate fractions of Pb bound to hydrogen sulfide and sulfide

In order to determine the impact of the enzyme on water samples that were originally digested with nitric acid, we compared the signal response both with and without enzyme addition. Figure 1 indicates that at high doses of acid, the enzyme has limited impact on the quantity of sample digested but at lower doses of acid, the enzyme provides an additional amount of digestion while also protecting the sensor from the impact of strong acid.
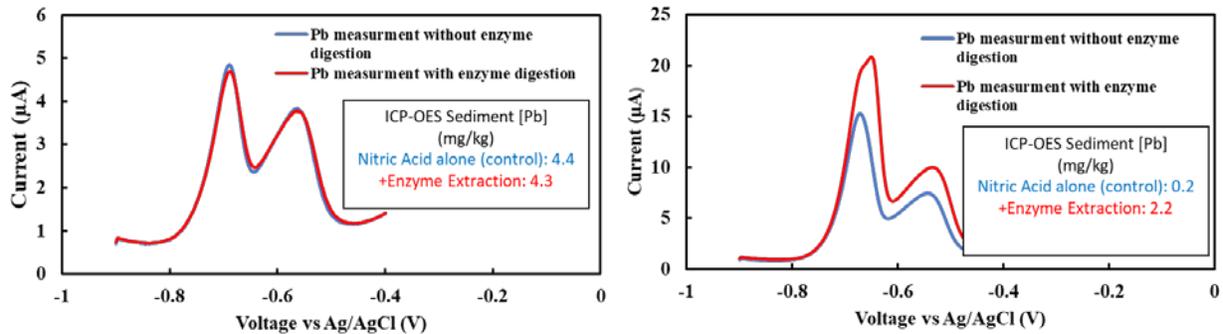
Figure 1. **Square wave anodic stripping voltammogram of lead ion in digested sediment sample with and without enzyme following with acidic digestion in 0.1 M nitric acid. Analysis was performed in 0.05 M nitric acid/ 0.1 M acetate buffer solution.**

2.5 Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) – Method Overview

Sediment sample preparation and analysis using inductively coupled plasma optical emission spectroscopy (ICP-OES) followed a modified version of US EPA Solid Wastes (SW) – 846 method 6010c. This method is prescribed for determination of 31 elements in ground water, industrial and organic wastes, soils, sludges and sediments following the use of a toxic leaching procedure (TCLP) or extraction procedure (EP) of digestion prior to analysis. As only a single element, Pb was of interest, precautions for interference were not taken during analysis. However, quality (calibration) and method performance were assessed with the use of calibration blanks, continuing calibration standards, and method blanks. Method blanks were less than 3 times the Pb detection limit determined by the calibration curve (calibration blanks and standards) of a given run. Continuing calibration standards were run every 8 to 10 samples and were used to correct Pb concentrations if values varied more than 10% of actual standard values. Method blanks were run for each step of 3-step sequential extraction procedure and for the total sediment digest to ensure low background Pb levels during acid digestions. Quality control measures were taken to run each step of the sequential sediment digest in duplicate to assess variability of Pb within a given digest step.

2.6 Apparatus and Materials – ICP-OES Analysis
- Ultra-high purity (UHP) nitrogen gas
- 15 mL Falcon centrifuge tubes, polypropylene

2.7 Reagents – ICP-OES
All analyses were performed using trace metal certified Pb standards diluted from concentrated (1000 mg/L) stock.

- 1 mg/L Lead nitrate (PbNO$_3$), in 3% nitric acid (HNO$_3$).

- Ultrapure (>17 megaohm) water

2.8 Protocol – ICAP-7400 ICP-OES Duo – Serial Number: IC74DC142110

*The instruction below are a summary of operational procedures given by Rutgers University – Department of Environmental Sciences, Environmental Chemistry Lab, Room 205C. Any further details regarding analysis can be found within the stated document.

Sample Preparation

1. All samples must be at least 5 mL in volume and free of particles (filtered or high-speed centrifugation)
2. HCl and HNO_3 concentrations must be less than 10% or sulfuric acid (H_2SO_4) less than 1%
3. Organic carbon concentrations must not exceed 10 mg/L

Instrument Preparation

- Release argon to instrument at 90 psi allowing Ar to flow for 30 to 60 minutes
- Turn on chiller and verify temperature reaches 20°C
- Ensure rinse bottle is filled with at least 1L of 2% (TMG) HNO_3 acid
- Ultrapure water is needed in first position of standard rack to rinse the nebulizer pre- and post-sample source of water for nebulizer drain sensor
- Set-up peristaltic pump using directions in operational procedures
- Flip power switch for autosampler, a green light will appear
- Create a LabBook before lighting torch to save Ar and rinse acid

Method/Results File – Qtegra

- Open Qtegra software and click LAB BOOK tab
- Make a new file name by either Create new from existing or Create new from a blank template then click Create LabBook
- Select elements from the periodic table
- Axial or radial measurement mode are available, for analysis of Pb, select axial mode as wavelengths generated by Pb are less than 230 nm
- Exposure times for UV and visible lines are defaulted to 15 and 5 s respectively
- To acquire spectral image capture, choose Capture, Full Frame, Yes to capture images of selected (see Sample list below) standards or samples or intelliframe to capture images of all standards and samples
- Exposure/integration times for UV and visible wavelengths for image to 2 to 5 s

Acquisition Parameters

- Analysis mode: normal is recommended or speed
- Pump speed = 50 rpm and flush pump speed = 100 rpm
- Stabilization time = 10 s

- Width: default for emission subarray width is 13

Standards

- A stored standard file can be used for Pb analysis
- Making a standard file available to other users by right clicking and select Save to global

Quantification

- Select fit type: linear or quadratic and intercept forcing as no, zero, blank
- Upper and lower limits to be flagged (control parameters) can be set in this section

Cetac ASX-520 Autosampler (if not selected, presently in manual mode)

- Open rack size (5x12)
- Wash and uptake times = 30 s

Autosampler Rack

- Load 15 mL tubes containing blanks, standards, and samples into racks
- Click +Add to add any rows for every blank, standard, and unknown
- Type the sample, standard, and blank names into table
- Select sample type (blk, std, unknown)
- Standard samples require selection of the standard file used allowing with dilution factors for standards
- Indicate correct rack number and vial position (located in far right column of sample list)
- If desired, select samples and/or standards for image capture under Full Frames column
- To save LabBook (select small folder in upper left hand corner) and customize export format as .csv spreadsheet

Starting the Plasma

- Select DASHBOARD and instrument picture in upper right corner to ensure the system is in autosampler mode (e.g. "iCAP 7400 w Cetac ASX520"); cancel if OK
- Click Cetac ASX-520 autosampler window and choose autosampler racks (Rinse draws nitric acid and Home location is above nitric acid
- Select "get ready" button to illuminate plasma and start peristaltic pump
  - Verify spectrometer optimization and ensure autosampler positions are go to vial, blank to rinse, After run: to vial Std rack 1 (QH_2O)
  - Optimization: carbon used for calibration in axial mode; offset values (x, y) are shown after the plasma lights and must be less than $\pm$ 3; otherwise the spec is out of calibration

- o Select "OK"
- Once plasma is lit, all Interlock indicators must be green and pump will begin *after optimization is finished*;

Check gas and system parameters

- Defaults: Auxiliary gas (-0.5 L/min), torch gas (12 L/min), nebulizer gas (0.5 L/min), pump speed (50 rpm)
- Gas flow for purge: Trickle (1.2 L/min, pause in work), normal (3.2 L/min, warm up), boost (5 L/min)
- Ensure proper orientation and tightness of peristaltic tubes (see operational procedure instructions)
- Manual operation of pump: type flow rate, TAB on keyboard, select "apply"
- In the case the torch assembly has been altered, undergo torch alignment with 2 ppm Zinc standard

3.0 Results

The samples used for this analysis were collected from Cheesequake Creek in NJ. They were digested using sequential extraction and accelerated enzyme digestion techniques. The results indicate that while the accelerated enzyme digestion cannot completely digest all of the lead in the sediment sample, the quantity that is digested can be used to predict the actual concentration using the sensor readings.

3.1 Lead Concentrations in Cheesequake Sediment

Lead concentrations for the sediment samples from Cheesequake Creek were measured using the standard EPA technique that applies strong acid in sufficient excess to digest the entire sample, the sequential extraction describe in the Method section, and the accelerated enzyme digestion process also described in the Method section of this report. Table 1 shows the measured concentrations for each of these procedures.

Table 2 Measured [Pb] using Digestion Protocols

Sample	Total [Pb] (Strong Acid Digestion)	Total [Pb] (Sequential Digestion)	Total [Pb] (Accelerated Enzyme Digestion)
SC1A	125	190	12
CC2B	100	125	8
CC7B	17	100	6
CC5A	63	17	3
SC2A	190	63	4

It is apparent from these data that the accelerated enzyme digestion cannot provide for a complete digestion of the lead contained in the sediment sample. Using the data, a correction method was investigated to determine if a simple multiplicative correction factor could be used to predict in-situ lead based on the Accelerated Enzyme Digestion results. The slope of the trend line in Figure 2 indicates that a multiplier of 15.579 can be applied to account for incomplete digestion.
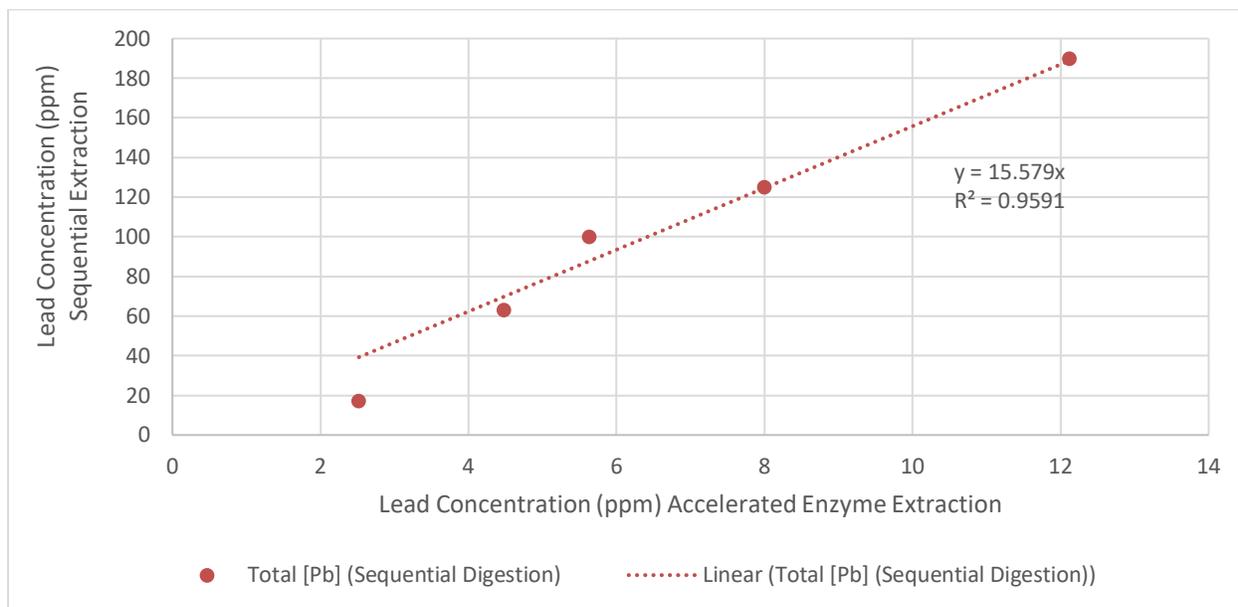
Figure 2 . Lead Concentration via Accelerated Enzyme Extraction and Sequential Extraction

3.2 Lead Fractionation in Cheesequake Sediment

Using the procedures outlined in the Method section of this report, the Pb concentration in the various Pb binding fractions of the sediment was determined. The results shown in Table 2 indicate that the majority of the Pb bound up in these samples can be found associated the Fe Oxides and Sulfides.

Table 3 Percentage of Pb Bound in four Sediment Fractions from Cheesequake Creek, New Jersey

Sample Name	F1- Carbonates % of Total	F2- Fe Oxides % of Total	F3- OM % of Total	F4- Residual Sulfides % of Total
SC2A	6.1	39.3	4.2	50.4
SC1A	3.1	34.6	15.9	46.4
CC2B	2.9	27.6	4.4	65.1
CC7B	4.4	40.1	ND	55.5
CC5A	9.6	40.7	4.7	45.0

In addition, a geochemical model was developed to determine each fraction based on the MEMs Sensor reading. The model was built to see both if it is possible to predict the fractionation of the Pb into the chemical fractions of the environment, and also if it is possible to improve upon the correction factor developed in the previous section. The process of building this model involved a stepwise series of regression models that were used to predict each fraction based the measured concentration of the Accelerated Enzyme Digested sediment.

The first equation in the geochemical model calculates the Pb concentration bound to the carbonate and organic matter fractions (F1+F3) of the sediment and is given by

$$F1+F3[Pb] = 14.6 + 2.57 * \ln(\text{In-Situ}[Pb]) \qquad [\text{Eq. 1}]$$

Equation 1 was derived by measuring the Pb bound to the F1+F3 fraction in Cheesequake sediments and plotting it against total sediment concentration as determined using the MEMs sensor, as shown in Figure 3. The modeled F1+F3-associated concentrations of Pb are shown in Table 3 next to the measured concentrations. The percent difference between the modeled and measured values ranged between 22% - 64%.
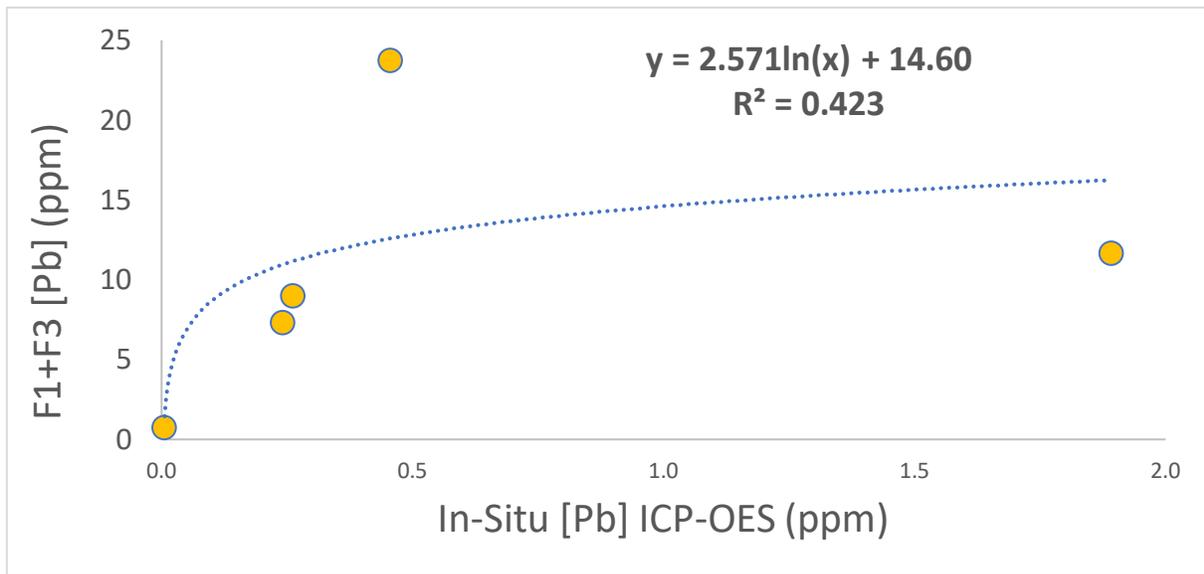
Figure 3 In-situ [Pb] post sediment-enzyme digest (x-axis) measured by ICP-OES vs. combined [Pb] in fraction 1 (F1) and fraction 3 (F3) (y-axis). Nonlinear regression (dashed lines) are F1+F3[Pb] = 14.60 + 2.571ln(In-Situ[Pb]) (R2 = 0.423).

Table 4 [Pb] ppm measured in in-situ sediment post sediment-enzyme digest by sediment-sensor and measured and modeled [Pb] in the combined F1+F3 fraction.

Sample	In-situ Dig. (Sensor) (ppm)	F1 + F3 Pb Fraction (ppm)	Modeled F1+F3 [Pb] (ppm)
SC2A	1.89	11.64	16.24
SC1A	0.46	23.71	12.58
CC2B	0.24	7.29	10.95
CC7B	0.01	0.75	1.45
CC5A	0.26	8.99	11.16

The second equation in the sediment geochemical model calculates the Pb concentration bound to the residual sulfide fractions (F4) of the sediment based on the calculated concentration of F1+F3-associated Pb. This fraction is calculated as

$$F4[Pb] = -6.39 + 28.2 * \ln(F1+F3[Pb]) \qquad [Eq. 2]$$

Equation 2 was derived by measuring the Pb bound to the F4 fraction and plotting it against the calculated Pb concentration bound to the F1+F3 fraction as calculated using Equation 1. The results are plotted in Figure 4. The modeled sulfide-associated concentrations of Pb were calculated using Equation 2 and are given in Table 4. For the sulfide fraction, the variation between the modeled and measured values was higher than for the carbonate fraction and ranged between 5% - 91%.
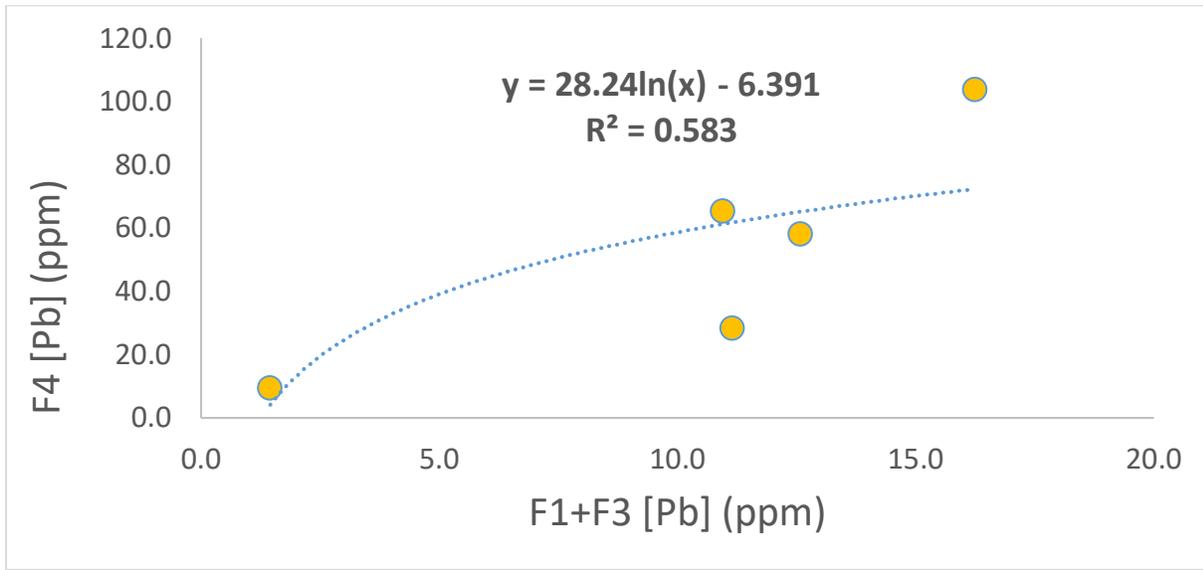

Figure 4 Modeled combined F1+F3[Pb] (x-axis) vs. [Pb] in fraction 4 (F4) (y-axis). Nonlinear regression (dashed lines) are F4[Pb] = -6.391 + 28.241ln(F1+F3[Pb]) (R2 = 0.583).

Table 5 [Pb] ppm measured in combined F1+F3 fraction by and measured and modeled [Pb] in the F4 fraction.

Sample	Modeled F1+F3 [Pb]	Sulfide (Res.) Pb Fraction – F4	Modeled Sulfide [Pb]
SC2A	16.24	103.61	68.99
SC1A	12.58	58.03	64.35
CC2B	10.95	65.15	61.83
CC7B	1.45	9.44	25.07
CC5A	11.16	28.34	62.17

The F4-associated (sulfide) Pb concentration can be used to calculate the iron oxide associated fraction (F2). In the sampled sediments, this was determined to be the fraction associated with the largest Pb concentration. Plotting the measured Pb bound to the F2 fraction and against the calculated F4 Pb concentration yields Equation 3.

$$F2[Pb] = -5.95 + 18.9 * \ln(F4[Pb]) \qquad [Eq.\ 3]$$

Once again, the modeled F2-associated concentrations of Pb were calculated and are presented with the measured concentrations in Table 5. The measured and calculated Pb concentrations in the iron oxide fraction exhibited a similar variability as in the sulfide fraction (1%-95%), with the exception of CC7B, which had a difference of 156%. This one sample had the highest relative percent difference in each step of the model which was compounded in successive calculations.
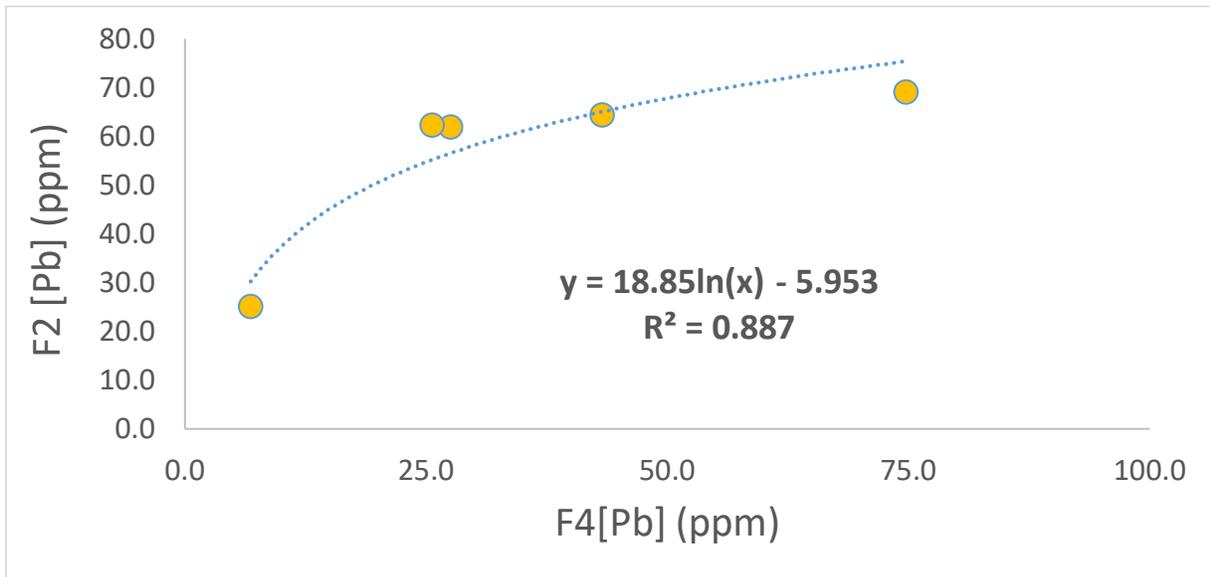
Figure 5 Modeled F4 [Pb] (x-axis) vs. measured fraction 2 (F2) (y-axis). Nonlinear regression (dashed lines) are F2[Pb] = -5.953 + 18.85ln(F4[Pb]) (R2 = 0.887).

Table 6 Modeled [Pb] in the combined F1+F3 fraction and modeled and measured and [Pb] in the F2 fraction.

Sample	Modeled Sulfide [Pb] (ppm)	Reducible Pb Fraction - F2 (ppm)	Modeled Reducible [Pb] (ppm)
SC2A	68.99	74.75	73.86
SC1A	64.35	43.26	72.55
CC2B	61.83	27.56	71.79
CC7B	25.07	6.81	54.78
CC5A	62.17	25.67	71.89

The modeled and measured concentrations for all fractions can be summed to determine the total Pb concentrations in the digested sediment. These values are shown in Table 6 and have been compared to the ICP-OES measured concentrations using a relative percent difference (RPD) calculation for each sample. Similar to the findings for each fraction, most of the samples had variability between 17%-79% for the measured vs. modeled values. Sample CC7B was the outlier with an RPD of 131%, which was influenced by the large RPD in the residual sulfide fraction, as shown in Table 7.

Table 7 Comparison (relative percent difference (RPD)) between total measured and modeled [Pb] in Cheesequake sediment via Geochemical Fraction Model and Linear Regression Multiplier.

Sample	Total [Pb] Measured	Total [Pb] Modeled	Total [Pb] Predicted via Multiplier
SC2A	190	161	189
SC1A	125	150	125
CC2B	100	145	88
CC7B	17	81	39
CC5A	63	146	70

Table 8 Comparison (relative percent difference (RPD)) between measured and modeled [Pb] in each fraction for various Cheesequake sediment samples.

Sample	F1+3 - Carbonates	F2 - Oxides	Residual - Sulfides
SC2A	33%	8%	34%
SC1A	61%	39%	22%
CC2B	40%	77%	10%
CC7B	64%	115%	141%
CC5A	22%	83%	87%

The calculated relative percent differences (RPDs) for measured vs. modeled lead concentration in each fraction are shown in Table 7. For the Cheesequake samples used in this study, the fraction containing the carbonate and organic matter (F1+3) was small and did not impact the variability of modeled-predicted lead concentrations. The oxide and sulfide fractions had a larger impact on

the modeled results and in addition the differences between modeled and measured lead was greater. Therefore, there is some evidence that the relative fractions in a sediment sample might impact the effectiveness of the enzyme, and consequently the model prediction.

4.0 Conclusions

The ability to perform a rapid digestion for the purpose of determining the concentration of Pb in sediment samples was tested. It was confirmed that the digested sample could be quantified equally well by both the standard ICP-OES and the novel MEMs sensor. The digestion was completed using an Accelerated Enzyme Digestion step that, while not providing complete digestion as is accomplished via other digestion procedures, was capable of digesting a relatively constant fraction of the Pb in the sediment sample. The digestion procedure detailed in section 2 of this report will be followed in subsequent phases of this sensor development. A geochemical model was also developed to predict the individual fractions of Pb found associated with the four chemical species identified in Table 2. The model provided acceptable results but was less accurate than a simple linear regression.

This work shows that a digestion step of relatively short duration (on the order of minutes) can be employed to replace the EPA digestion procedure that uses strong acids and takes 24 hours. The next steps in the development of this sensor system include additional measurements over a wide range of sediment samples and development and testing of a prototype to enable sediment sample analysis.

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