Geosyntec consultants

A Best Practices User's Guide for Sediment Porewater Passive Sampling for Inorganic Constituents of Concern

Jason Conder, Florent Risacher, Elizabeth Nichols, Haley Schneider, Michaela Lawrence (Geosyntec Consultants)

Brent G. Pautler, Alex Sweett (SiREM)

Gunther Rosen (NIWC Pacific)

Andrew Jackson (Texas Tech University)

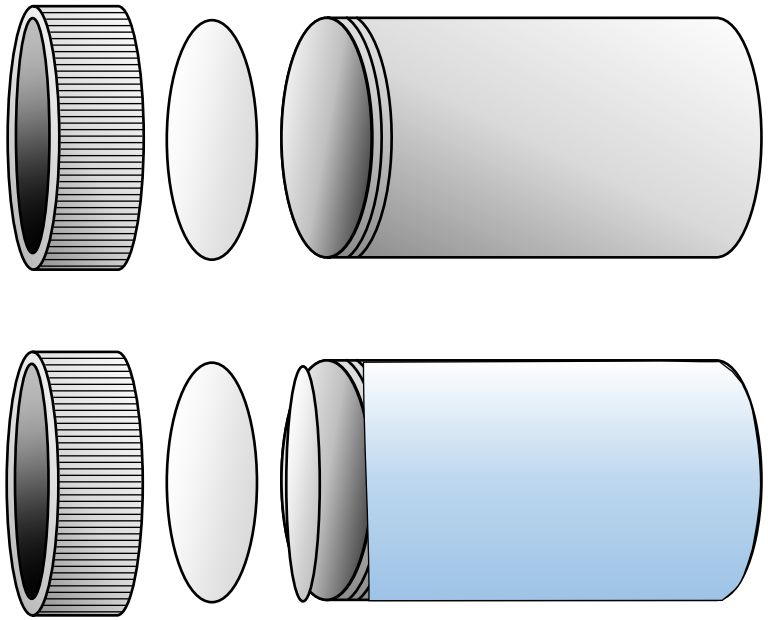
November 13, 2024
Sediment Management Work Group Sponsors Meeting, Spring, TX

A User's Guide for Inorganics Passive Sampling

- Best Practice User's Guide (BPUG) for using passive samplers for measuring the availability of inorganic constituents (primarily metals) in sediment and water
  - Dialysis passive samplers, aka "peepers"
  - Primary audience is consultants and regulators working at contaminated sediment sites
  - Also applicable to researchers in environmental chemistry, ecotoxicology, remediation, etc.

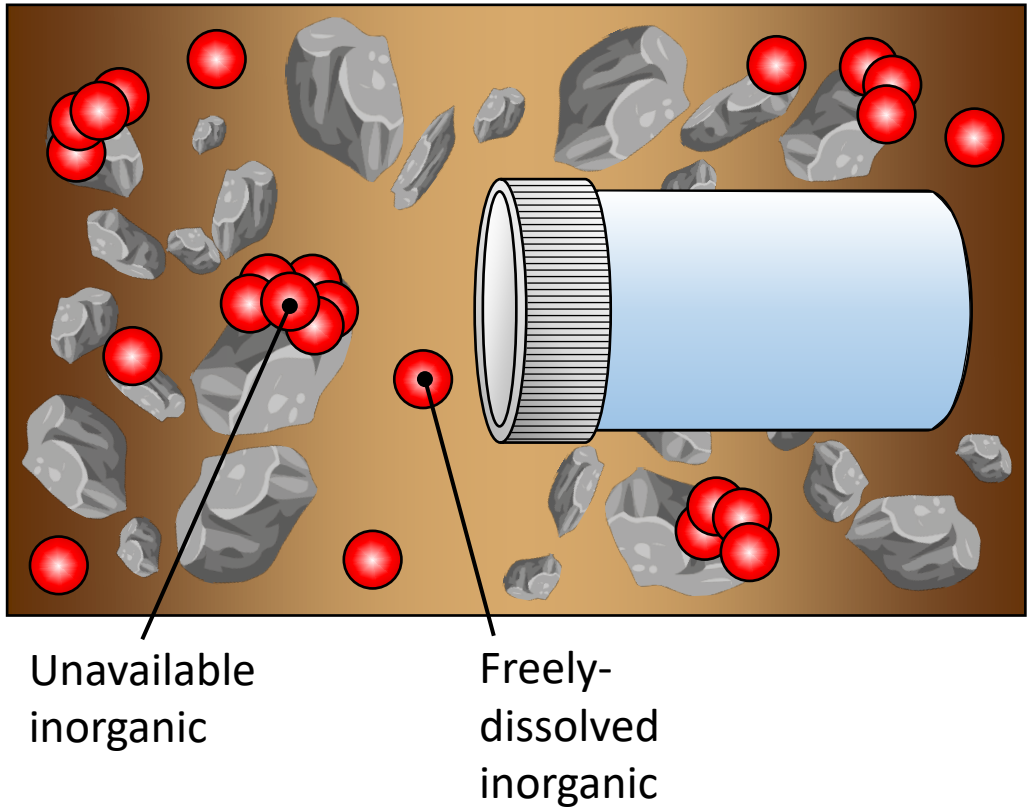
Available now at the above link (I hope!) or email jconder@geosyntec.com for a free PDF of the Best Practices User's Guide

Peepers



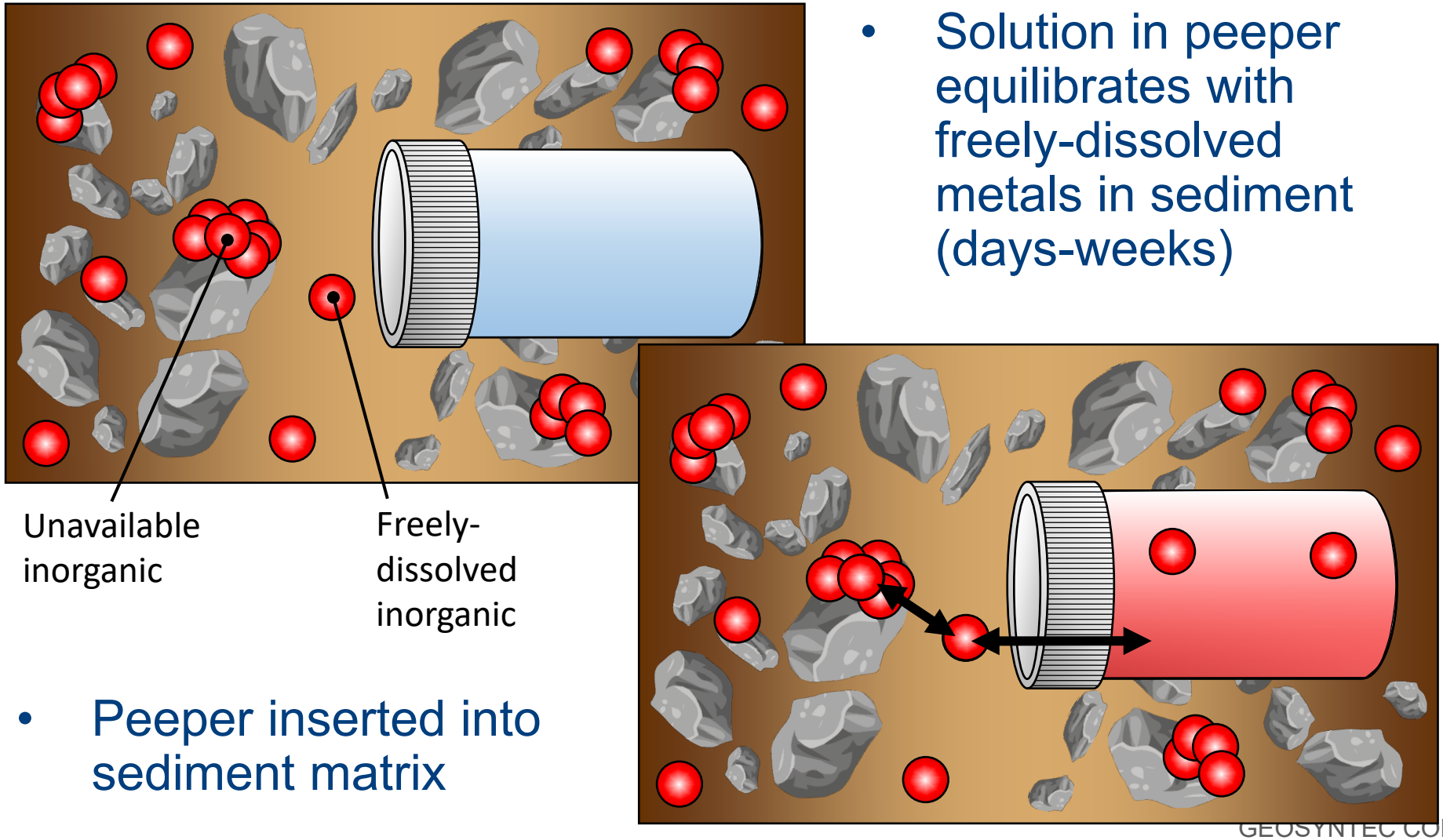
- Small container (chamber) capped with semi-permeable membrane
- Can also have protective outer cap (with open permeations)
- Filled with ultrapure water

Peeper Use in Sediment



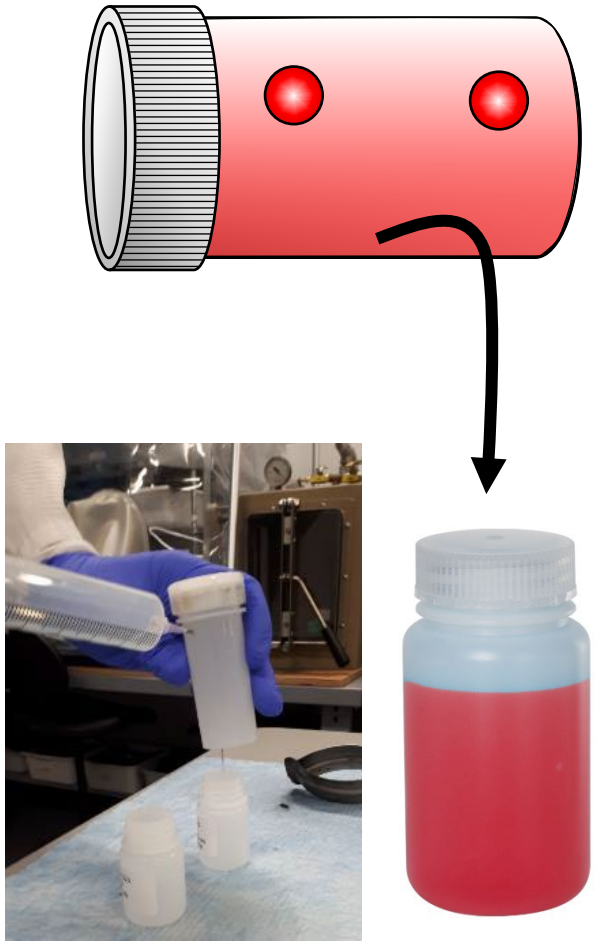
- Peeper inserted into sediment matrix

Peeper Use in Sediment

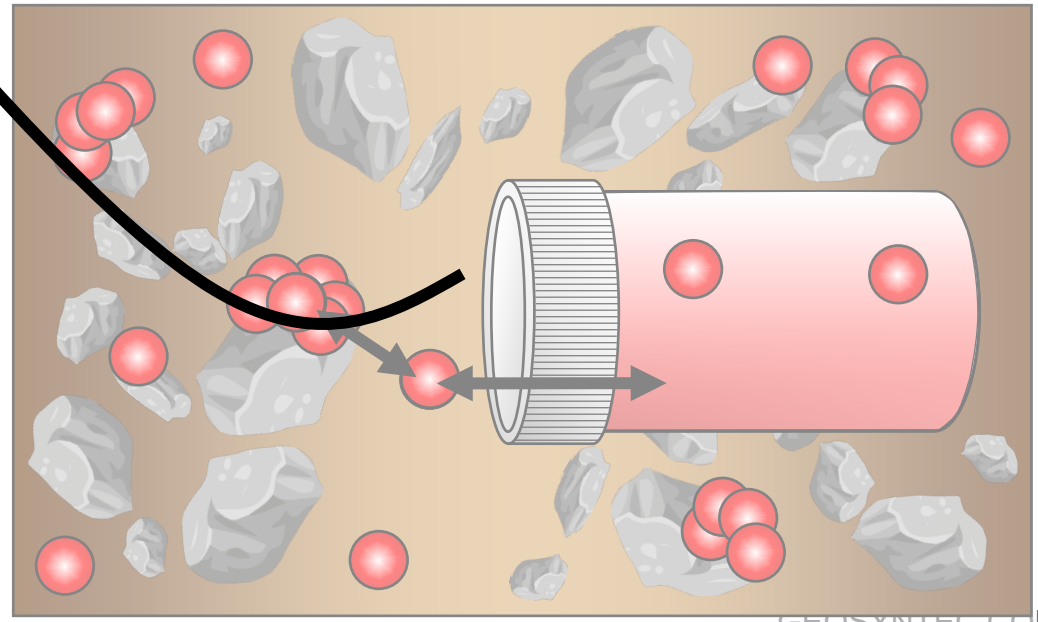


- Solution in peeper equilibrates with freely-dissolved metals in sediment (days-weeks)
- Peeper inserted into sediment matrix

Peeper Use in Sediment



- Peeper removed from sediment, solution transferred and preserved, measured for inorganic analytes using standard methods for water (e.g., EPA SW846)



Our Project

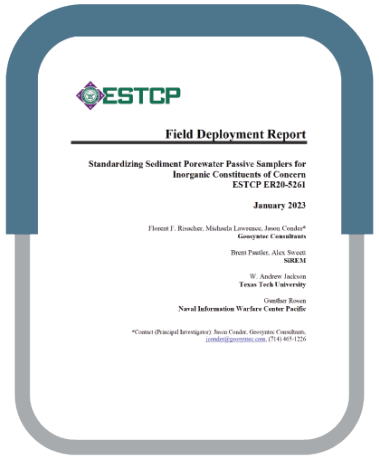
- Dialysis samplers (“peepers”) in use for nearly 50 years to understand metal availability at contaminated sediment sites, but there are a lack of standard methods
- Our 3-year ESTCP project (2021-2024): Optimization and standardization
  - ✓ Better peeper designs
  - ✓ Robust and streamlined methods
  - ✓ Procedures and guidance for end-user community
  - ✓ Field demonstration
  - ✓ Communication and technical transition

Best Practice's User's Guide

Best Practices User's Guide

Literature Review

Laboratory Testing



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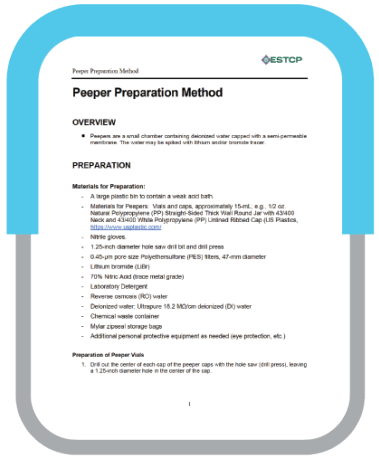

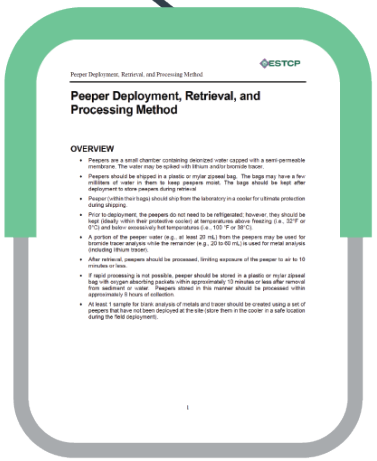

Standard Operating Procedures

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Best Practice's User's Guide

Best Practices User's Guide


ESTCP

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Standardizing Sediment Porewater Passive Samplers for Inorganic Constituents of Concern
ESTCP ER20-5261

November 2023

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Towards Standardizing Peeper Samplers for Sediment Inorganics

A Review of Methods and Data Gaps

SERDP Project: ER20-5261
April 2021

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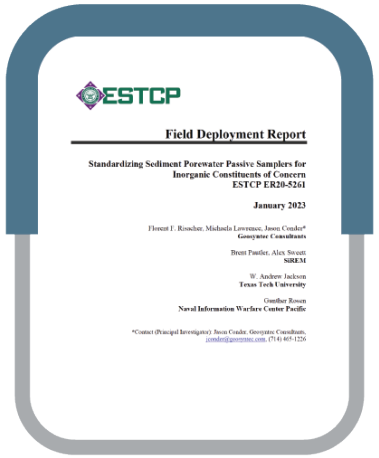
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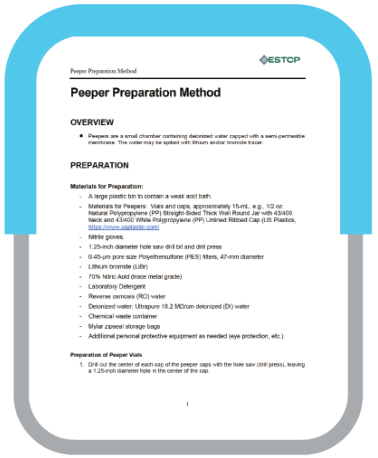
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Peeper Preparation Method

OVERVIEW

- Peepers are a small chamber containing deionized water capped with a semi-permeable membrane. The water may be spiked with lithium and/or bromide tracer.

PREPARATION

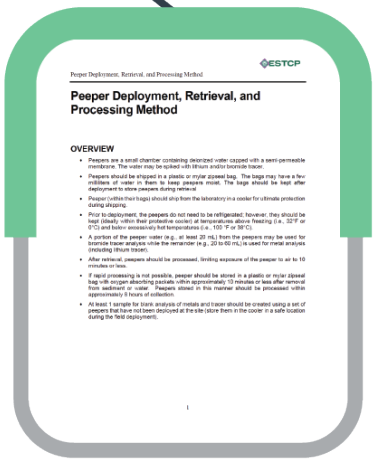
Materials for Preparation:

- A large plastic bin to contain a weak acid bath
- Materials for Peepers: Vials and caps, approximately 15-mL, e.g., 1/2 oz Natural Polypropylene (PP) Straight-Sided Thick Wall Round Jar with 43/400 Neck and 43/400 White Polypropylene (PP) Unlined Ribbed Cap (US Plastics, https://www.usplastic.com/)
- Nitrile gloves
- 1.25-inch diameter hole saw drill bit and drill press
- 0.45-µm pore size Polyethersulfone (PES) filters, 47-mm diameter
- Lithium bromide (LiBr)
- 70% Nitric Acid (trace metal grade)
- Laboratory Detergent
- Reverse osmosis (RO) water
- Deionized water: Ultrapure 18.2 MΩ/cm deionized (DI) water
- Chemical waste container
- Mylar zipseal storage bags
- Additional personal protective equipment as needed (eye protection, etc.)

Preparation of Peeper Vials

1. Drill out the center of each cap of the peeper caps with the hole saw (drill press), leaving a 1.25-inch diameter hole in the center of the cap.

Standard Operating Procedures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Peeper Deployment, Retrieval, and Processing Method

OVERVIEW

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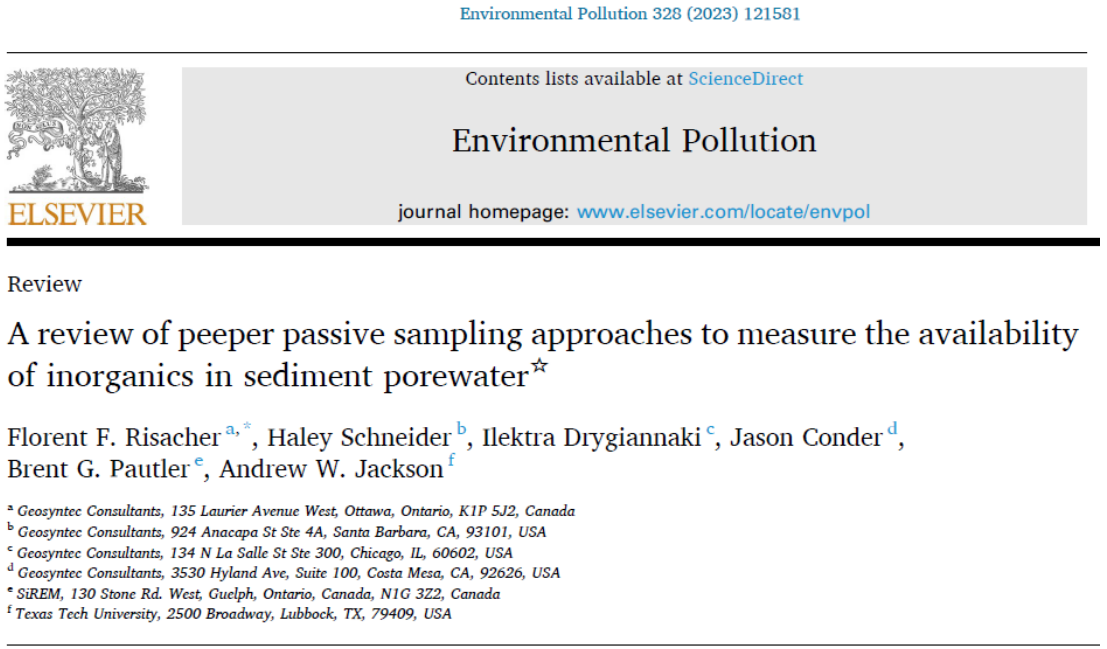
- Peeper (within their bags) should ship from the laboratory in a cooler for ultimate protection during shipping.
- Prior to deployment, the peepers do not need to be refrigerated; however, they should be kept (ideally within their protective cooler) at temperatures above freezing (i.e., 32°F or 0°C) and below excessively hot temperatures (i.e., 100 °F or 38°C).
- A portion of the peeper water (e.g., at least 20 mL) from the peepers may be used for bromide tracer analysis while the remainder (e.g., 20 to 60 mL) is used for metal analysis (including lithium tracer).
- After retrieval, peepers should be processed, limiting exposure of the peeper to air to 10 minutes or less.
- If rapid processing is not possible, peeper should be stored in a plastic or mylar zipseal bag with oxygen absorbing packets within approximately 10 minutes or less after removal from sediment or water. Peepers stored in this manner should be processed within approximately 8 hours of collection.
- At least 1 sample for trank analysis of metals and tracer should be created using a set of peepers that have not been deployed at the site (store them in the cooler in a safe location during the field deployment).

Part 1: Literature Review

- Review of > 85 peer reviewed papers on sediment passive sampling for inorganics from the past > 45 years
- Summary of what works, what doesn't, and what needs to be improved
- Key topics
  - Peeper design (material, membranes, chamber volumes and design factors)
  - Pre-equilibrium sampling methods
  - Oxygen contamination (during deployment, after deployment



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journal homepage: www.elsevier.com/locate/envpol

Review

A review of peeper passive sampling approaches to measure the availability of inorganics in sediment porewater☆

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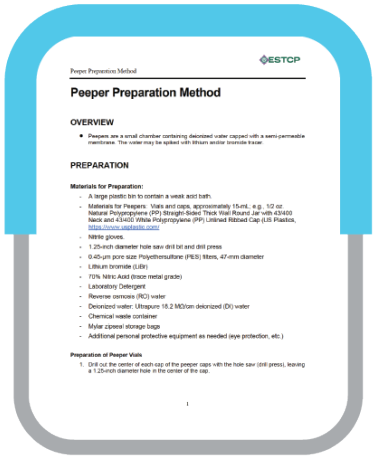
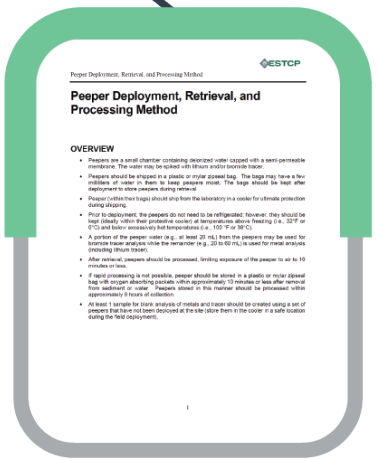




Standard Operating Procedures

Part 2: Lab Studies

1. Peeper Design

- 60 mL HDPE
- 20 mL PTFE
- 15 mL PP
- Choose best peeper design and optimal deployment time for remaining experiments

2. Deoxygenation

- Deoxygenated vs. non-deoxygenated peepers
- Evaluate effect of oxygen on concentration of inorganics

3. Peeper Water Transfer

- Process in air using simple and inert gas transfer methods and compare to nitrogen box
- Identify optimal peeper water transfer method

4. Peeper Shelf Life

- Evaluate oxygen content of peeper water over time in stored peepers
- Evaluate sample storage approach and time period prior to processing

5. Post-Deployment Storage Time

- Store peeper in various storage scenarios to evaluate oxygen contamination
- Evaluate sample storage approach and time period prior to processing

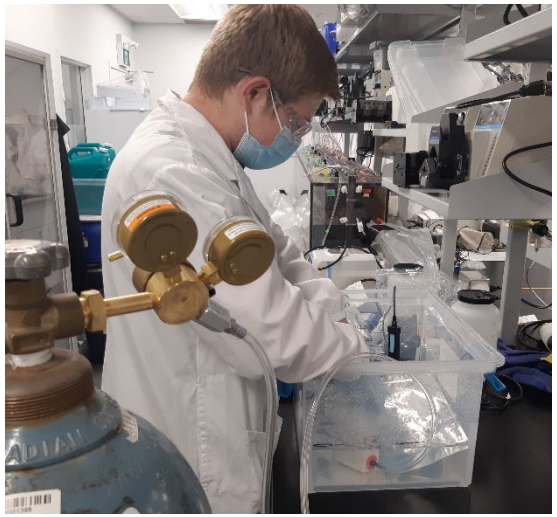
6. Pre-equilibrium Sampling Approach

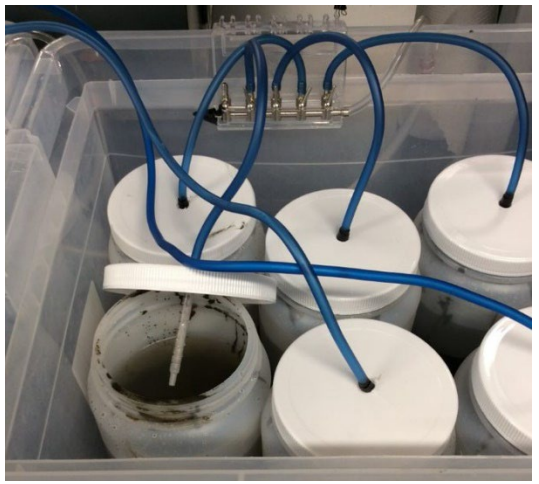
- Deploy in test sediment for multiple durations
- Demonstrate and validate reverse tracer for pre-equilibrium deployments

7. Peeper Water Salinity Effects

- Evaluate standard deionized and saline peeper water
- Evaluate effect of initial peeper water salinity on pre-equilibrium sampling approach in marine sediment

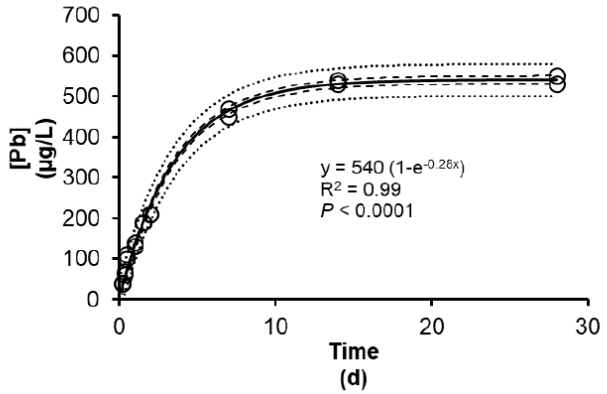
- Conducted with a standard marine test sediment
  - 3.8-L HPDE jar
  - ~1.2 kg, ww sediment
- Overlying water aerated synthetic marine water
- 14-day peeper deployments (unless otherwise specified)

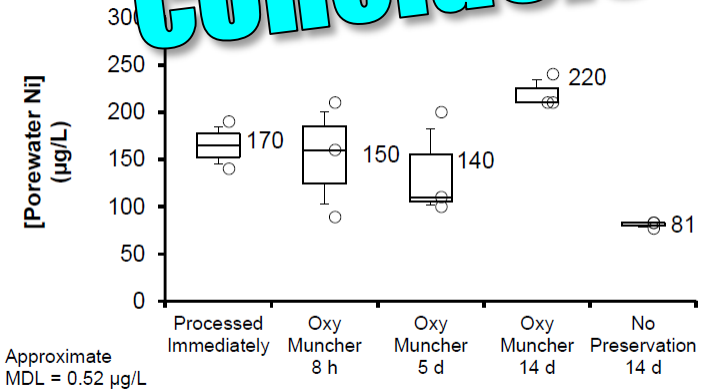


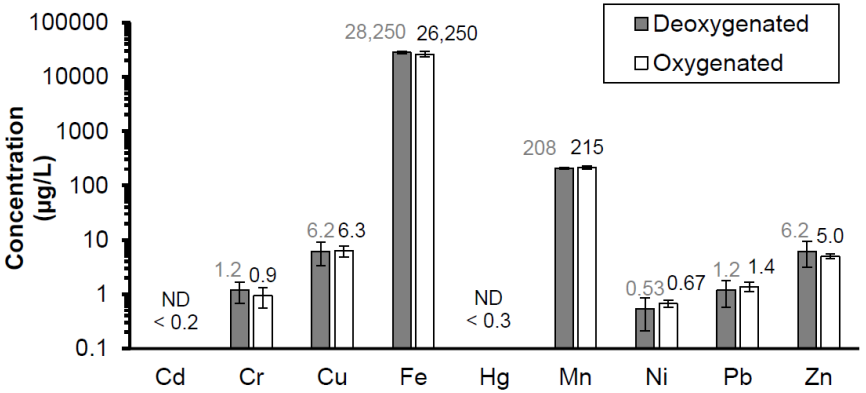


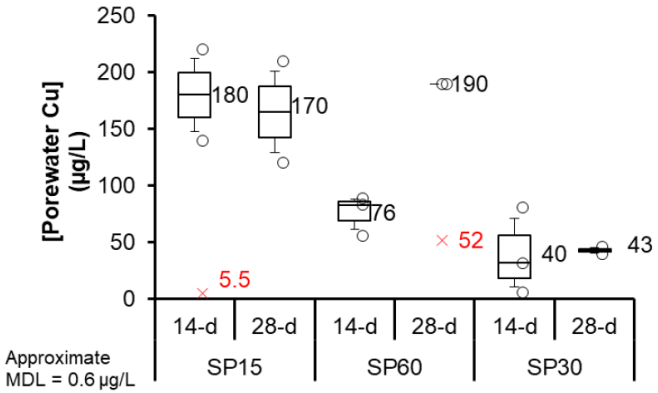
Part 2: Lab Studies

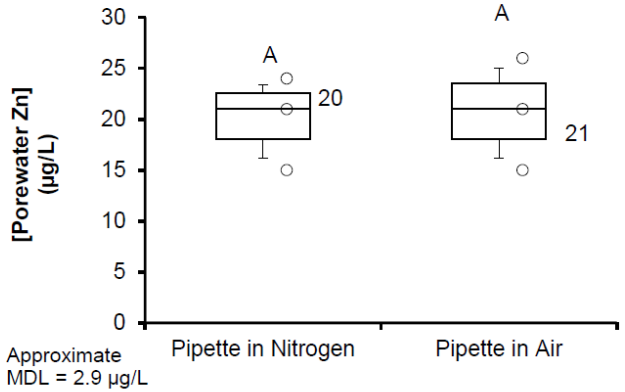
All sorts of neat lab data & conclusions

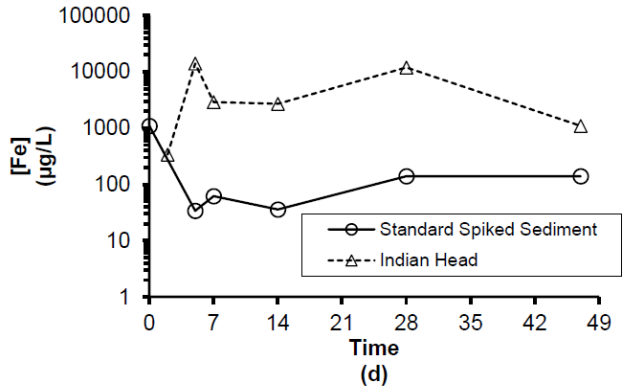












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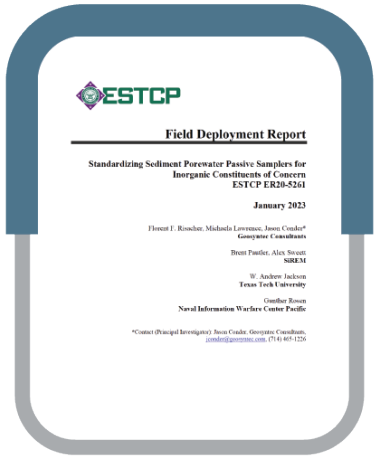
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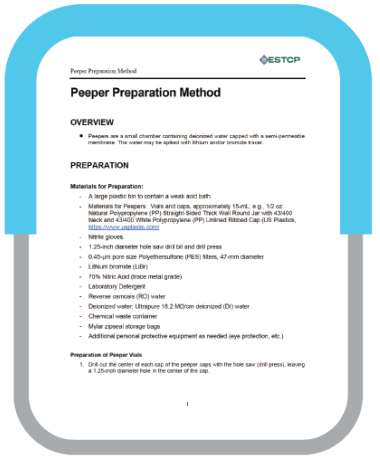
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Peeper Preparation Method

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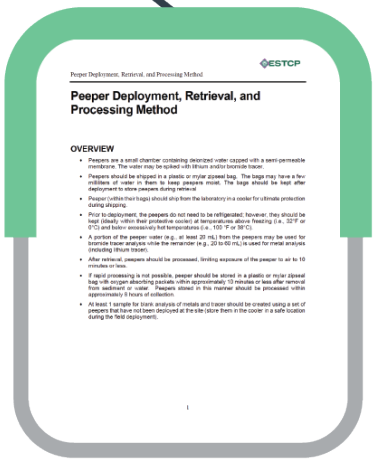
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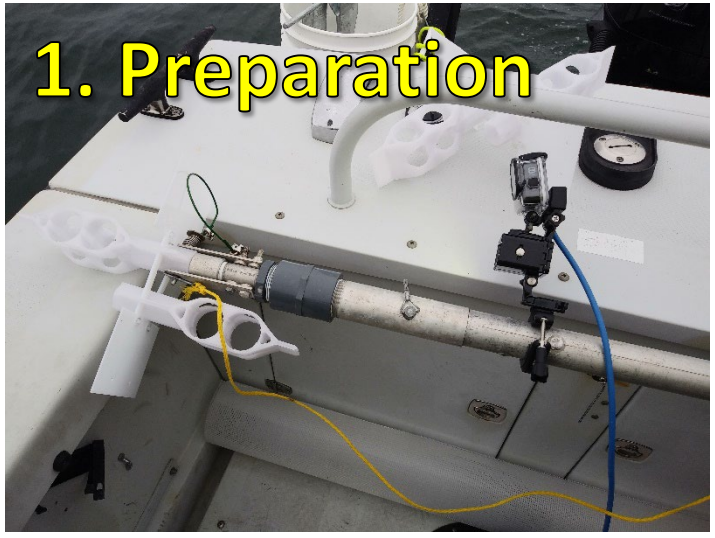
- Peeper (within their bags) should ship from the laboratory in a cooler for ultimate protection during shipping.
- Prior to deployment, the peepers do not need to be refrigerated; however, they should be kept (ideally within their protective cooler) at temperatures above freezing (i.e., 32°F or 0°C) and below excessively hot temperatures (i.e., 100 °F or 38°C).
- A portion of the peeper water (e.g., at least 20 mL) from the peepers may be used for bromide tracer analysis while the remainder (e.g., 20 to 60 mL) is used for metal analysis (including lithium tracer).
- After retrieval, peepers should be processed, limiting exposure of the peeper to air to 10 minutes or less.
- If rapid processing is not possible, peeper should be stored in a plastic or mylar zipseal bag with oxygen absorbing packets within approximately 10 minutes or less after removal from sediment or water. Peepers stored in this manner should be processed within approximately 6 hours of collection.
- At least 1 sample for trank analysis of metals and tracer should be created using a set of peepers that have not been deployed at the site (store them in the cooler in a safe location during the field deployment).

Part 3: Field Demonstration + Standard Operating Procedures (SOPs)

- Field deployment of peepers in mouth of Paleta Creek, Naval Base San Diego
- Push-pole deployment and retrieval, 10 stations, 10 days in October 2022
- Analysis of metals

Part 3: Field Demonstration + Standard Operating Procedures (SOPs)



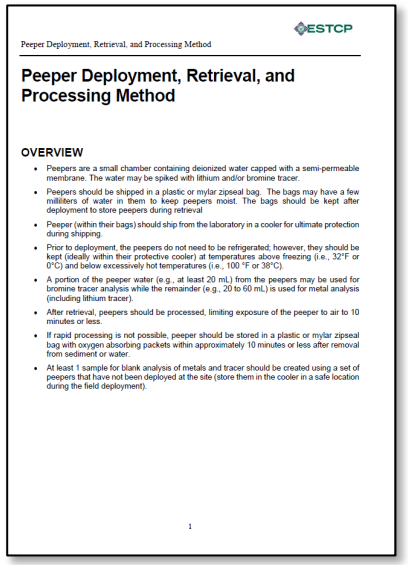
- Deployment video

Part 3: Field Demonstration + Standard Operating Procedures (SOPs)

- Retrieval video
- Processing video

Part 3: Field Demonstration + Standard Operating Procedures (SOPs)

- Practical tools for investigators
  - Step-by-step deployment, retrieval, and processing Standard Operating Procedures (SOPs)
  - Metadata on personnel logistics and timing for field work (time per station for deployment, retrieval, processing, etc.)
  - Easy-to-use Excel tool (attached to our field report) for tracer data calculations



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- At least 1 sample for blank analysis of metals and tracer should be created using a set of peepers that have not been deployed at the site (store them in the cooler in a safe location during the field deployment).

1

Table 14: Preparation and Deployment Time

Sampling station	Vessel Positioning Time (minutes)	Push Pole Deployment Time (minutes)
1A	16	26
1B	0 (same location as 1A)	5
1C	0 (same location	
2	14	
3	37	
4	9	
5	9	
6	5	
7	7	
8	13	

Table 15: Retrieval Times for Day 2

Sampling station	Diver Retrieval Time (minutes)
1A	40*
1B	
1C	5
2	Retrieved on Day 1
3	15
4	10
5	30
6	30
7	30
8	-

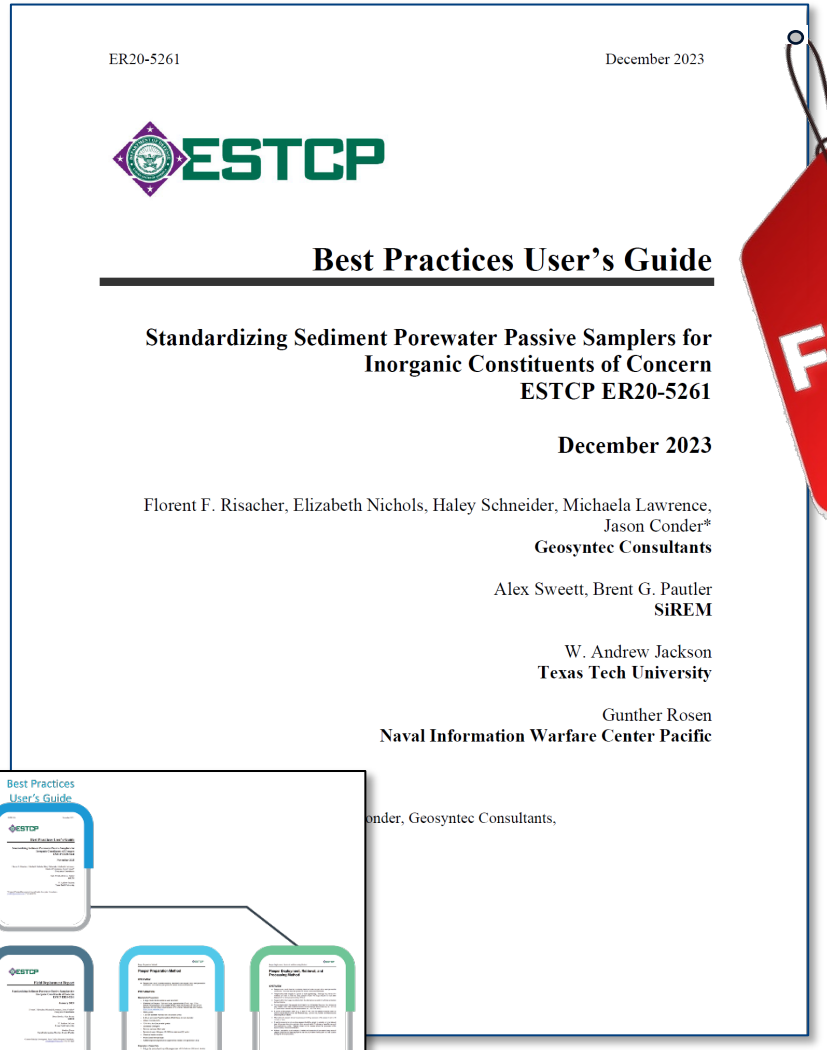
* Station 1A and 1 B were recovered together within 40 minutes.
- Station 8 was not retrieved.

Table A1. Calculation of CFree values using the lithium tracer, San Diego peeper samples.
Equilibrium Correction of Porewater Concentration using Lithium Tracer

Sample ID	Sample Deployment Date	Sample Collection Date	Chemical Name	Chemical Abreviation	Report Result Text	Measured Concentration in Peeper	MDL in Peeper	Report Result Unit	Lab Qualifiers	Initial Li Concentration (µg/L)	Deployment Time (days)	Assumed Li Concentration External to Peeper (µg/L)	Sample-Specific Elimination Rate for Li (K_{el}) (day^{-1})	Diffusion Coefficient for Analytes (Di) (* 10^{-5} cm^2/s)	Di + D_{EL}	Sample-specific Elimination Rate for Analytes (K_i) (day^{-1})	Percent Equilibrium Reached (%)	Equilibrium Corrected Concentration (µg/L)	Equilibrium Corrected MDL (µg/L)	Equilibrium Corrected Result (µg/L) 2-sig figs
1A-PW	10/3/22 12:30	10/14/22 14:58	Lithium	Li	16000	16000	26	µg/L		86000	11.10	100	0.152	0.90	1.00	0.152	81	--	--	--
1A-PW			Cadmium	Cd	ND	0	0.27	µg/L			11.10			0.63	0.70	0.107	69	0.00	0.39	**< 0.39**
1A-PW			Chromium	Cr	ND	0	1.9	µg/L			11.10			0.52	0.58	0.088	62	0.00	3.05	**< 3**
1A-PW			Copper	Cu	3.2	3.2	1.4	µg/L			11.10			0.62	0.69	0.105	69	4.65	2.04	**4.7**
1A-PW			Iron	Fe	220	220	35	µg/L			11.10			0.63	0.70	0.107	69	317.23	50.47	**320**
1A-PW			Lead	Pb	0.88	0.88	0.21	µg/L	J		11.10			0.83	0.92	0.140	79	1.11	0.27	**1.1**
1A-PW			Manganese	Mn	25	25	1.7	µg/L			11.10			0.62	0.69	0.105	69	36.35	2.47	**36**
1A-PW			Mercury	Hg	ND	0	0.13	µg/L			11.10			0.74	0.82	0.125	75	0.00	0.17	**< 0.17**
1A-PW			Nickel	Ni	ND	0	0.65	µg/L			11.10			0.62	0.69	0.105	69	0.00	0.95	**< 0.95**
1A-PW			Zinc	Zn	13	13	3.6	µg/L			11.10			0.61	0.68	0.103	68	19.07	5.28	**19**
1A-SW	10/3/22 12:30	10/14/22 14:58	Lithium	Li	150	150	1.4	µg/L		86000	11.10	100	0.671	0.90	1.00	0.671	100	--	--	--
1A-SW			Cadmium	Cd	ND	0	0.36	µg/L			11.10			0.63	0.70	0.470	99	0.00	0.36	**< 0.36**
1A-SW			Chromium	Cr	20	20	2.6	µg/L			11.10			0.52	0.58	0.388	99	20.27	2.64	**20**
1A-SW			Copper	Cu	13	13	1.9	µg/L			11.10			0.62	0.69	0.463	99	13.08	1.91	**13**
1A-SW			Iron	Fe	110	110	46	µg/L			11.10			0.63	0.70	0.470	99	110.60	46.25	**110**
1A-SW			Lead	Pb	0.36	0.36	0.28	µg/L	J		11.10			0.83	0.92	0.620	100	0.36	0.28	**0.36**
1A-SW			Manganese	Mn	12	12	2.2	µg/L			11.10			0.62	0.69	0.463	99	12.07	2.21	**12**
1A-SW			Mercury	Hg	ND	0	0.33	µg/L			11.10			0.74	0.82	0.552	100	0.00	0.33	**< 0.33**
1A-SW			Nickel	Ni	29	29	0.86	µg/L			11.10			0.62	0.69	0.463	99	29.17	0.87	**29**
1A-SW			Zinc	Zn	23	23	4.8	µg/L			11.10			0.61	0.68	0.455	99	23.15	4.83	**23**
1B-PW	10/3/22 13:25	10/14/22 14:56	Lithium	Li	510	510	0.83	µg/L		86000	11.06	100	0.483	0.90	1.00	0.483	100	--	--	--
1B-PW			Cadmium	Cd	ND	0	0.22	µg/L			11.06			0.63	0.70	0.339	98	0.00	0.23	**< 0.23**
1B-PW			Chromium	Cr	ND	0	1.5	µg/L			11.06			0.52	0.58	0.280	95	0.00	1.57	**< 1.6**
1B-PW			Copper	Cu	4.8	4.8	1.1	µg/L			11.06			0.62	0.69	0.333	97	4.92	1.13	**4.9**
1B-PW			Iron	Fe	4400	4400	28	µg/L			11.06			0.63	0.70	0.339	98	4506.30	28.68	**4500**
1B-PW			Lead	Pb	0.39	0.39	0.17	µg/L	J		11.06			0.83	0.92	0.446	99	0.39	0.17	**0.39**
1B-PW			Manganese	Mn	230	230	1.3	µg/L			11.06			0.62	0.69	0.333	97	235.91	1.33	**240**
1B-PW			Mercury	Hg	ND	0	0.13	µg/L			11.06			0.74	0.82	0.398	99	0.00	0.13	**< 0.13**
1B-PW			Nickel	Ni	1	1	0.52	µg/L			11.06			0.62	0.69	0.333	97	1.03	0.53	**1**
1B-PW			Zinc	Zn	20	20	2.9	µg/L			11.06			0.61	0.68	0.328	97	20.55	2.98	**21**

Conclusions: Best Practices User's Guide Available Now

- Best Practices User's Guide ties everything together from the extensive 3-year lit review, lab, and field efforts
  - User-friendly guide that enables end-users to successfully prepare, deploy, and retrieve peepers, as well as interpret and use peeper data in a decision-making context at sediment sites under regulatory oversight
  - Written as a list of 17 Frequently Asked Questions (FAQs)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ESTCP

Best Practices User's Guide

Standardizing Sediment Porewater Passive Samplers for Inorganic Constituents of Concern
ESTCP ER20-5261

December 2023

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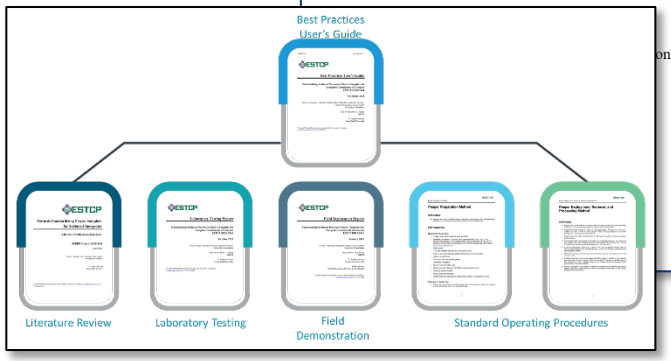


FAQs at A Glance (1 of 2)

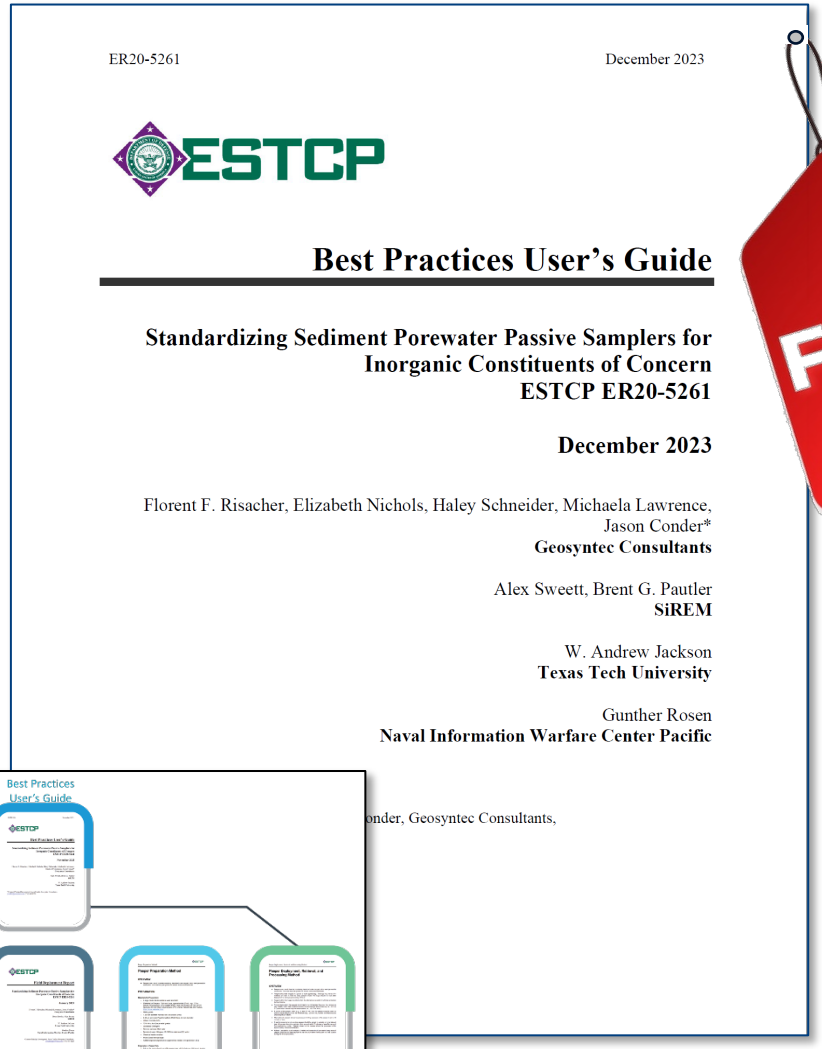
FAQ	Main Answers
Why measure metal availability in sediment?	• Allows an improved understanding of fate, risks, and remediation performance
What is a peeper, and how does it measure metal availability?	• Peepers are simple passive samplers that accumulate metals into a water sample via diffusion from the sediment or water in which the peeper is deployed
Are there other abiotic tools to measure metal availability?	• AVS/SEM analysis of bulk sediment and DGT passive samplers are also popular tools
Where can peepers be obtained?	• Commercial service providers and academic/government research laboratories
How are peepers prepared?	• Peepers are cleaned, filled with ultrapure water, and capped with a semipermeable membrane (see Appendix D for an example SOP)
Do peepers sample colloidal or other sorbed metals from sediment?	• Limited research suggests peepers do not sample these phases, and inorganic analytes that pass through peeper membranes are assumed to represent "dissolved" species
Do peepers and peeper water need to be deoxygenated prior to deployment in sediment?	• No – the presence of oxygen in peeper water at the time of deployment does not affect results
Does the peeper water salinity need to be the same as the salinity of the water or sediment in which it is to be deployed?	• No – peepers should be prepared with deionized water, even when deployed in marine or estuarine environments
How are peepers deployed in sediment or water?	• A variety of methods can be used (wading, from a vessel, using SCUBA divers, etc.) • See Appendix C for an example of peeper deployment, this how-to video, and Appendix E for an example deployment SOP
How long are peepers left to equilibrate in sediment and water?	• Typically a few days to a few weeks – it depends on the size of the peeper and its membrane, and whether the peeper is deployed in sediment or water • Pre-equilibrium approaches using tracers can be used to interpret peeper data when peepers are deployed for a period that is not sufficient to attain full equilibration

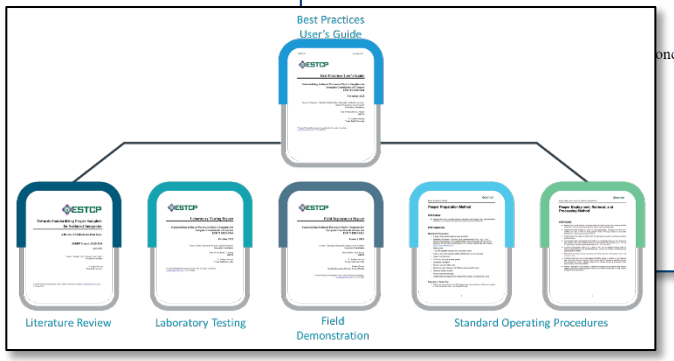
FAQs at A Glance (2 of 2)

FAQ	Main Answers
Does biofouling affect peepers?	• Usually not if deployment is only a few days or weeks
How are peepers retrieved from sediment or water?	• Using SCUBA divers, a grappling hook, or other methods • See Appendix C for an example of peeper retrieval, this how-to video, and Appendix E for an example retrieval SOP
When and how do you process retrieved peepers?	• Transfer of peeper water into storage containers provided by the commercial analytical laboratory should be completed within 8 hours of retrieval • Processing can be conducted in air – it does not need to be conducted in an inert atmosphere • See Appendix C for an example of peeper processing, this how-to video, and Appendix E for an example processing SOP
How are the peeper samples analyzed and what detection limits can be attained?	• Peeper water can be analyzed using any standard method • Detection limits depend on the minimum volume of water specified by the method
How can peeper data be validated?	• Standard data validation and QA/QC approaches for sediment and surface water sampling and analysis can be easily adapted to peepers
How are peeper data used at a sediment site?	• Peeper data can help quantify availability, nature and extent, and potential aquatic life toxicity of inorganics in sediment and water
What is the cost of a peeper investigation?	• Peepers represent an additional cost for most sediment investigations, but the high value of the data and its ability to reduce uncertainty provides a high return on the investment. • Hypothetical all-inclusive costs for an example 20-peeper investigation ranged from approximately $60K to $150K

Conclusions: Best Practices User's Guide Available Now

- Best Practices User's Guide ties everything together from the extensive 3-year lit review, lab, and field efforts
  - User-friendly guide that enables end-users to successfully prepare, deploy, and retrieve peepers, as well as interpret and use peeper data in a decision-making context at sediment sites under regulatory oversight
  - Written as a list of 17 Frequently Asked Questions (FAQs)
  - **1-stop shopping for the whole project; attachments include:**
    - **117-page lab study**
    - **51-page field study**
    - **SOPs**
    - **Excel model files**
    - **Links to online how-to videos**

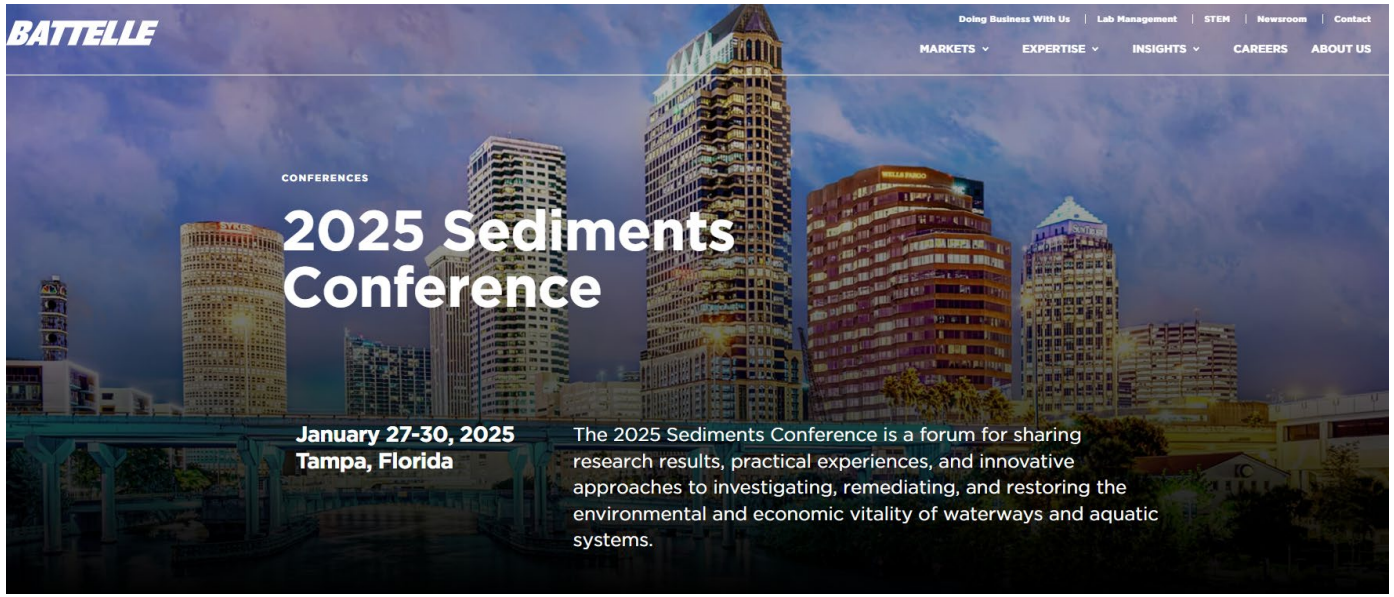




Conclusions: Best Practices User's Guide Available Now

One-day Only Technical Training Course

- 4-hour short course on using dialysis passive samplers for metals (deep dive on this project) and PFAS
- January 27, 2025 at the Battelle 2022 Sediments Conference, Tampa, FL
  - Sign up now, limited spaces available
  - Early bird short course registration savings by November 11



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Thank You for Listening

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