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The webinar will begin promptly at
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Approaches for Managing Contaminated Sediments

December 10, 2020

SERDP DOD • EPA • DOE | ESTCP

Welcome and Introductions

Rula A. Deeb, Ph.D.
Webinar Coordinator

SERDP DOD • EPA • DOE | ESTCP

Webinar Agenda

- **Webinar Logistics** (5 minutes)
  Dr. Rula Deeb, Geosyntec Consultants
- **Overview of SERDP and ESTCP** (5 minutes)
  Dr. Andrea Leeson, SERDP and ESTCP
- **Standardizing Polymeric Sampling for Measuring Freely-Dissolved Organic Contaminants in Sediment Porewater** (25 minutes + Q&A)
  Dr. Mandy Michalsen, U.S. Army Engineer Research Development Center
- **Long Term Monitoring of Activated Carbon Amendment in Sediments at an Active Shipyard** (25 minutes + Q&A)
  Mr. Gunther Rosen, Naval Information Warfare Center Pacific
- **Final Q&A session**

Zoom Instructions

- Download Zoom
  - https://zoom.us/download
- If you cannot download Zoom, you can view the slides using an internet browser
  - Create a free Zoom account (https://zoom.us/signup)
  - Use a compatible browser (Firefox, IE or Edge)
  - View the webinar at https://success.zoom.us/wc/95796329470/join
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- If you experience difficulties with the audio, call into the conference line
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  - Required webinar ID: 957-9632-9470
- We will also be live streaming the webinar on the SERDP and ESTCP YouTube channel
  - https://www.youtube.com/user/SERDPESTCP

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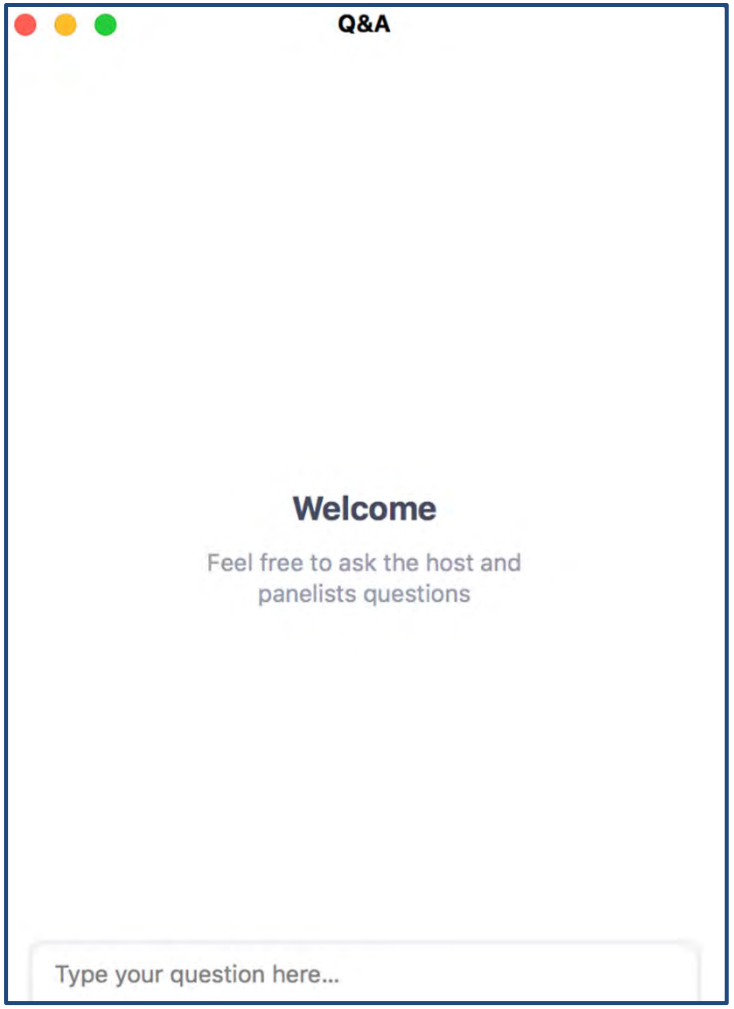
- Find the Q&A button on your control bar and type in your question(s)
- Make sure to add your organization name at the end of your question so that we can identify you during the Q&A sessions



SERDP and ESTCP Overview

Andrea Leeson, Ph.D.
SERDP and ESTCP

DoD's Environmental Technology Programs

Science and Technology

- Statutory program established 1991
- DoD, DOE, EPA partnership
  - Advanced technology development to address near-term needs
  - Fundamental research to impact real world environmental management

Demonstration and Validation

- Demonstrate innovative cost-effective environmental and energy technologies
  - Transition technology out of the lab
  - Establish cost and performance
  - Partner with end user and regulator
  - Technology transfer
    - Accelerate commercialization or broader adoption
    - Direct technology insertion

Environmental Drivers

Sustaining Ranges, Facilities and Operations

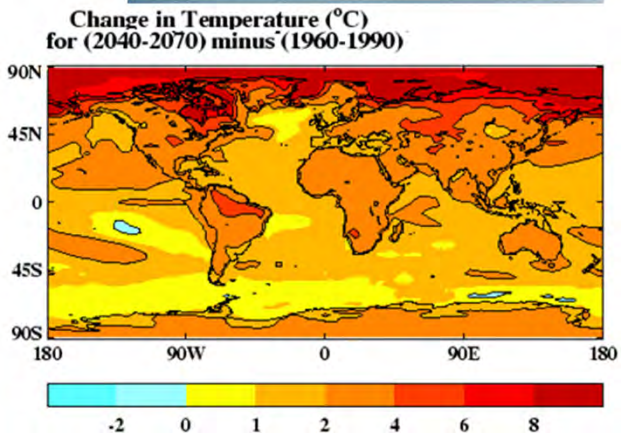
Maritime Sustainability
Threatened and Endangered Species

Toxic Air Emissions and Dust

UXO and Munitions Constituents

Noise

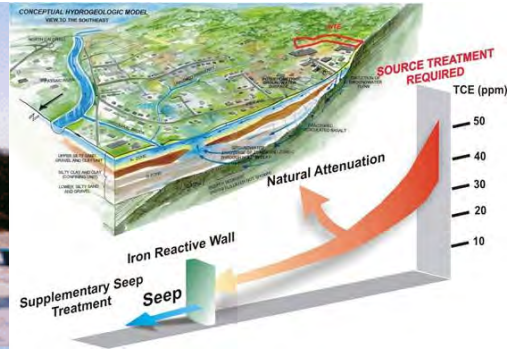
Sustainable FOB



Environmental Drivers

Reducing Current and Future Liability

Contamination from Past Practices



- Groundwater, soils and sediments
- Large UXO liability
- Emerging contaminants

Pollution Prevention to Control Life Cycle Costs

- Elimination of pollutants and hazardous materials in manufacturing, maintenance, and operations
- Achieve compliance through pollution prevention

www.serdp-estcp.org

SERDP and ESTCP Webinar Series

Date	Topic
January 14, 2021	Improving Energy Efficiency through Advanced Building Controls
January 28, 2021	Improved Approaches for PFAS Sampling and Treatment
February 11, 2021	Advances in the Detection of Submerged Unexploded Ordnance in Marine Environment
February 25, 2021	Managing Chlorinated Solvents in Groundwater Using Biological Treatment
March 11, 2021	New Resource Conservation Paradigms on DoD Lands
March 25, 2021	Safer Alternatives for Surface Engineering and Structural Materials in Weapons Systems and Platforms: A Fred Lafferman Tribute Webinar

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Standardizing Polymeric Sampling for Measuring Freely-Dissolved Organic Contaminants in Sediment Porewater

Mandy Michalsen, Ph.D., P.E.
U.S. Army Engineer Research Development Center

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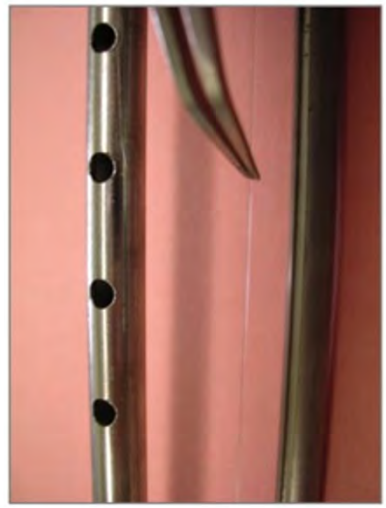
Agenda

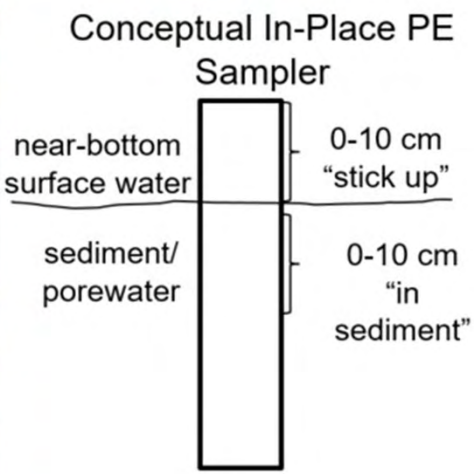
- Technology description
- Project objectives
- Results by task
- Conclusions
- Benefits to DoD
- Acknowledgments

What are Polymeric Samplers?

LDPE in a metal casing upon retrieval from sediment



PDMS surrounding a glass core



***Note**: LDPE = low-density polyethylene; PMDS = polydimethylsiloxane*

Objectives

- Develop standard operating procedures (SOPs)
- Use SOPs to complete interlab method validation study
  - Fulfill EPA SW-846 requirements for method application
- Technology transfer
  - Prepare instruction videos and webinars to promote standardized method

Task 1: Interlab Analytical Method Comparability

Verify acceptable instrumentation & analytical capabilities between labs

Calibration Check Standards Sent to Labs (e.g. NIST 1493 PCBs, NIST 1647f PAHs)	PAHs and PCBs ± 30% known concentrations

Collaboratively unify methods & identify performance reference compounds (PRCs)

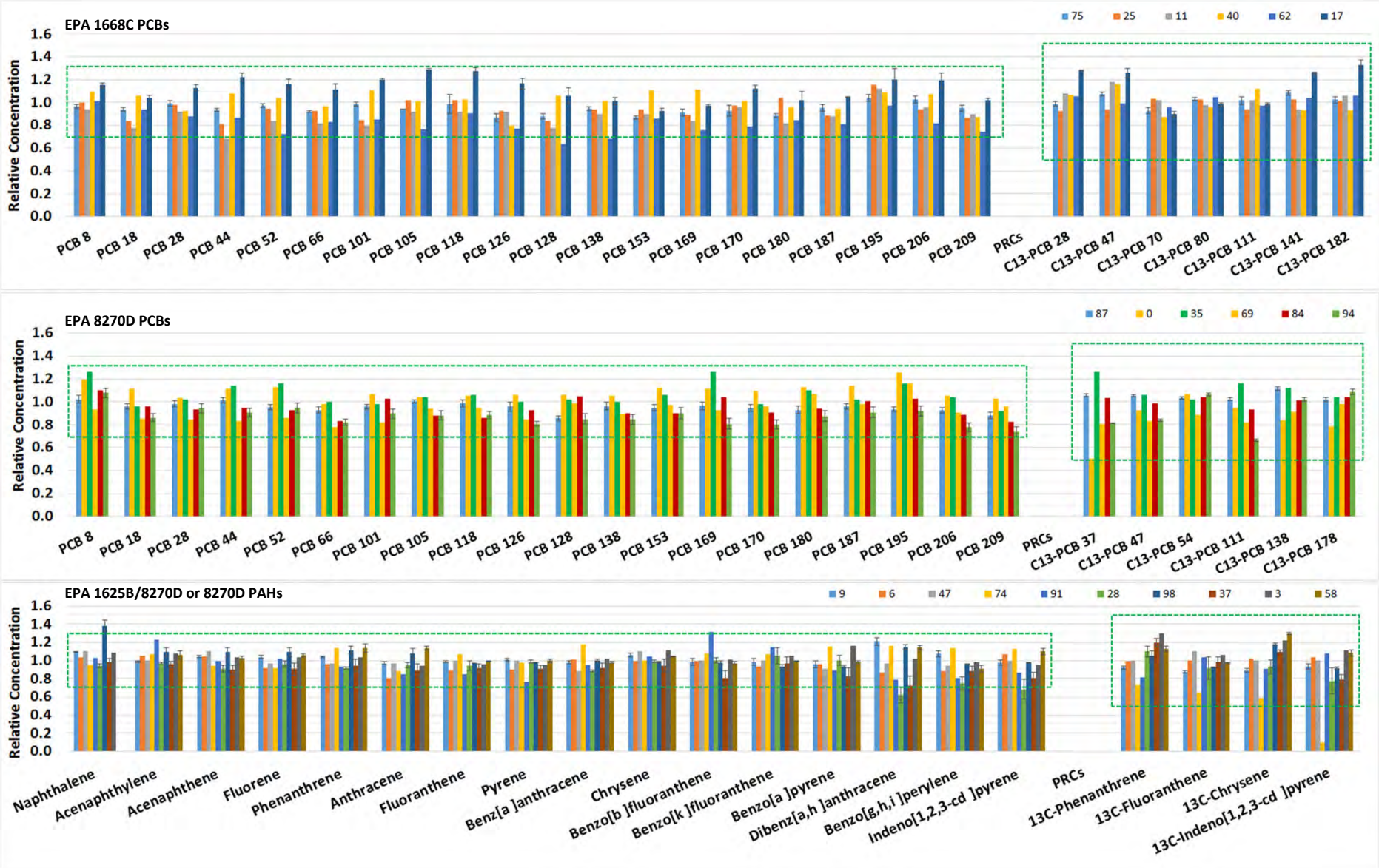
Select suite of PRCs for PAHs, PCBs acceptable to all participating labs	Finalize PRCs and methods for demonstration

PRC pre-loaded samplers

All commercial laboratories receive PRC-preloaded polymeric samplers for analysis by final methods

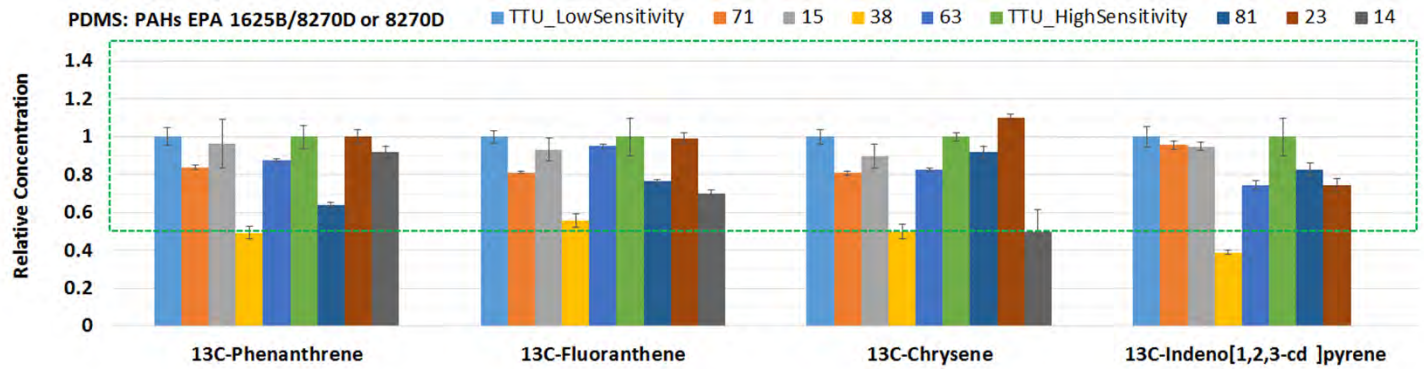
Labs analyze samplers	Accuracy: ±30% of known concentration* Precision: ±20% relative standard deviation*

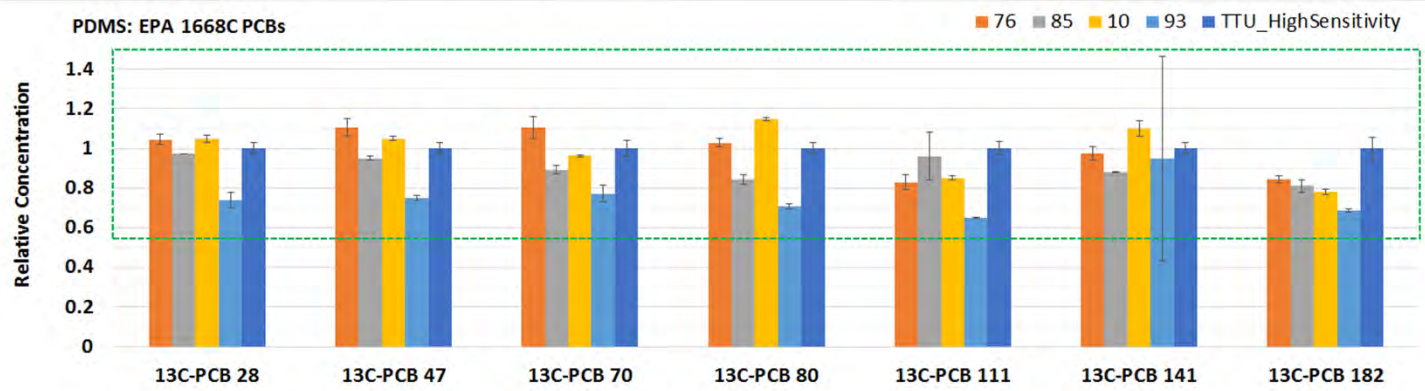
* Variation outside of control limits may be acceptable for one or more targets

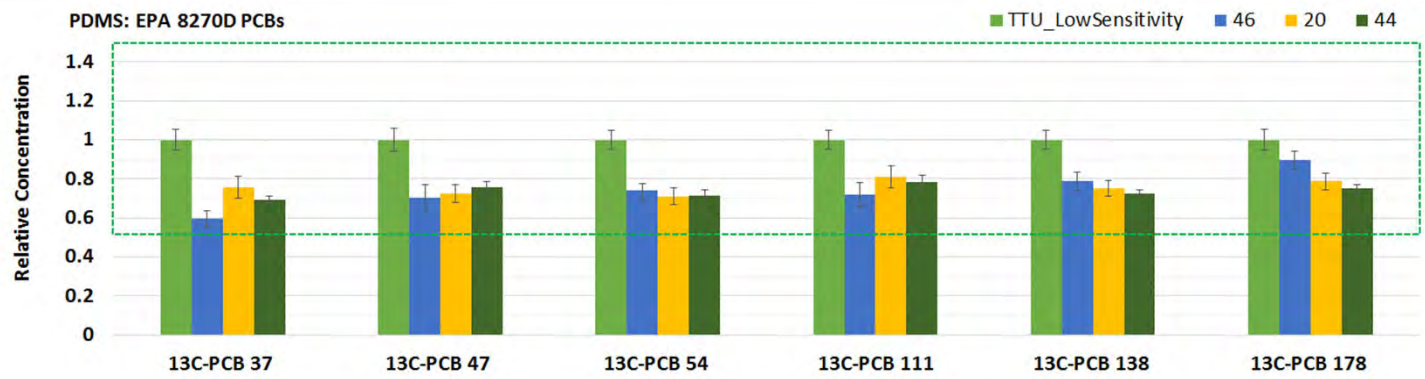


Calibration Check Standard Results: Labs Achieved ± 30% of Target

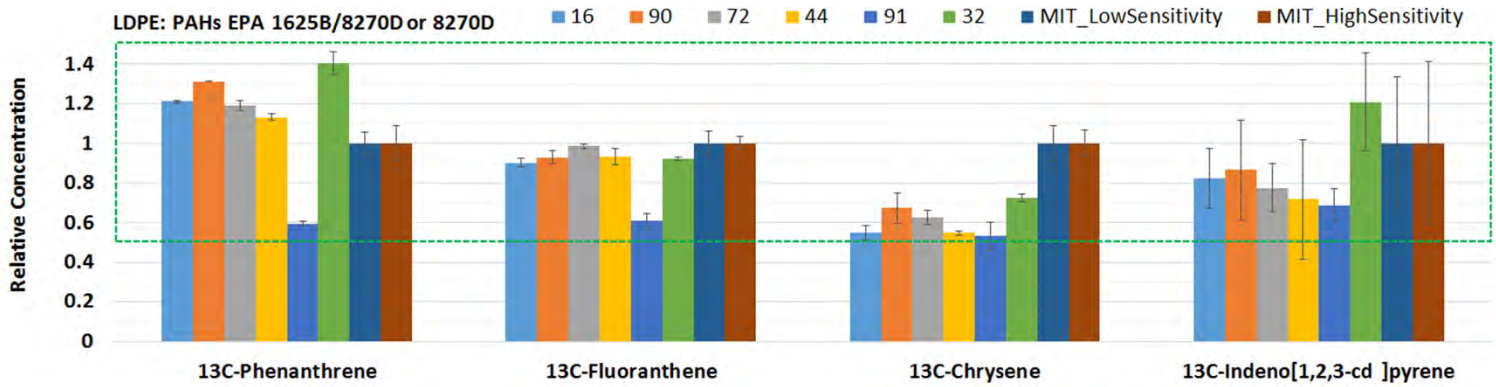
PDMS

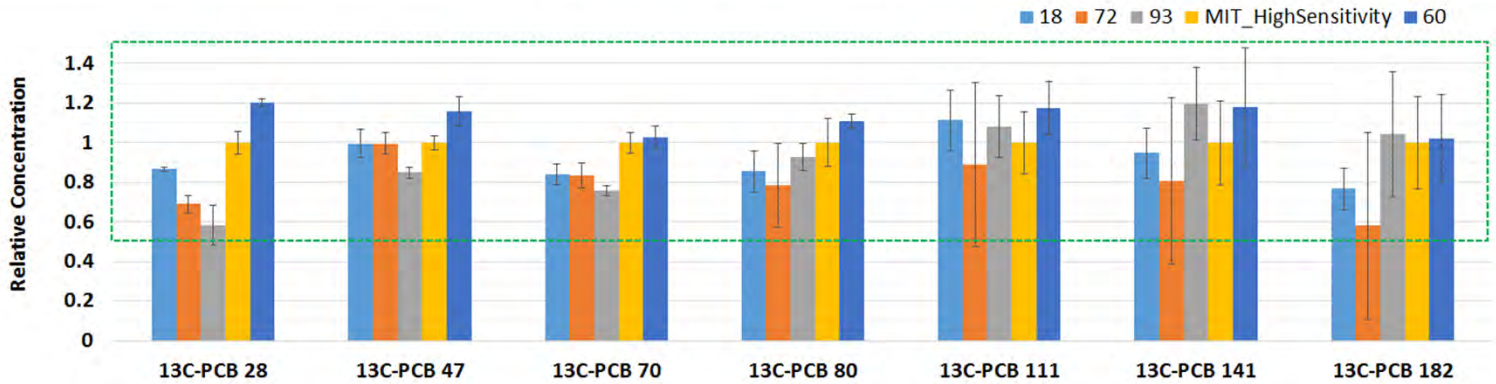

PDMS: PAHs EPA 1625B/8270D or 8270D
TTU_LowSensitivity
71
15
38
63
TTU_HighSensitivity
81
23
14
Relative Concentration
1.4
1.2
1
0.8
0.6
0.4
0.2
0
13C-Phenanthrene
13C-Fluoranthene
13C-Chrysene
13C-Indeno[1,2,3-cd]pyrene

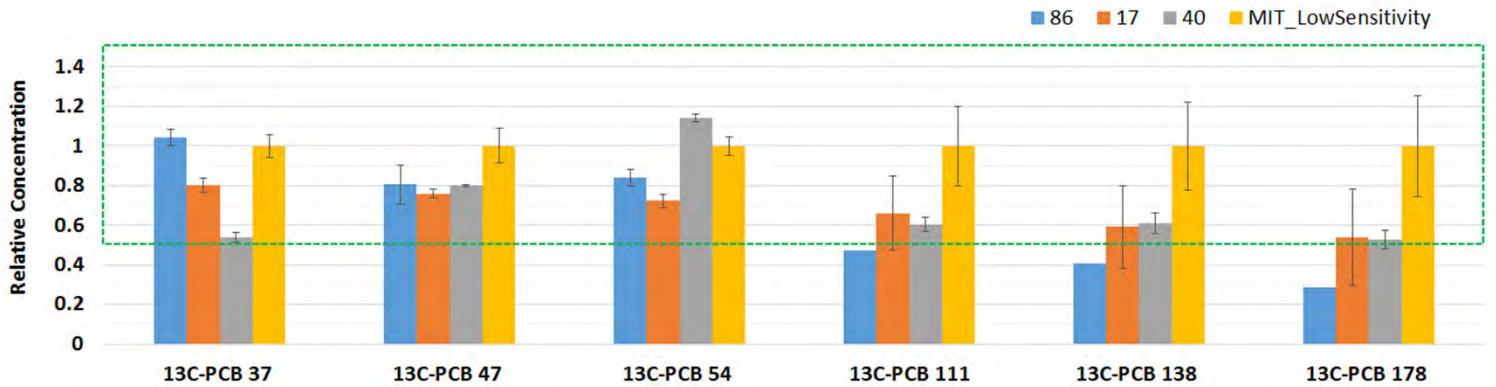

PDMS: EPA 1668C PCBs
76
85
10
93
TTU_HighSensitivity
Relative Concentration
1.4
1.2
1
0.8
0.6
0.4
0.2
0
13C-PCB 28
13C-PCB 47
13C-PCB 70
13C-PCB 80
13C-PCB 111
13C-PCB 141
13C-PCB 182


PDMS: EPA 8270D PCBs
TTU_LowSensitivity
46
20
44
Relative Concentration
1.4
1.2
1
0.8
0.6
0.4
0.2
0
13C-PCB 37
13C-PCB 47
13C-PCB 54
13C-PCB 111
13C-PCB 138
13C-PCB 178

LDPE


LDPE: PAHs EPA 1625B/8270D or 8270D
16
90
72
44
91
32
MIT_LowSensitivity
MIT_HighSensitivity
Relative Concentration
1.4
1.2
1
0.8
0.6
0.4
0.2
0
13C-Phenanthrene
13C-Fluoranthene
13C-Chrysene
13C-Indeno[1,2,3-cd]pyrene

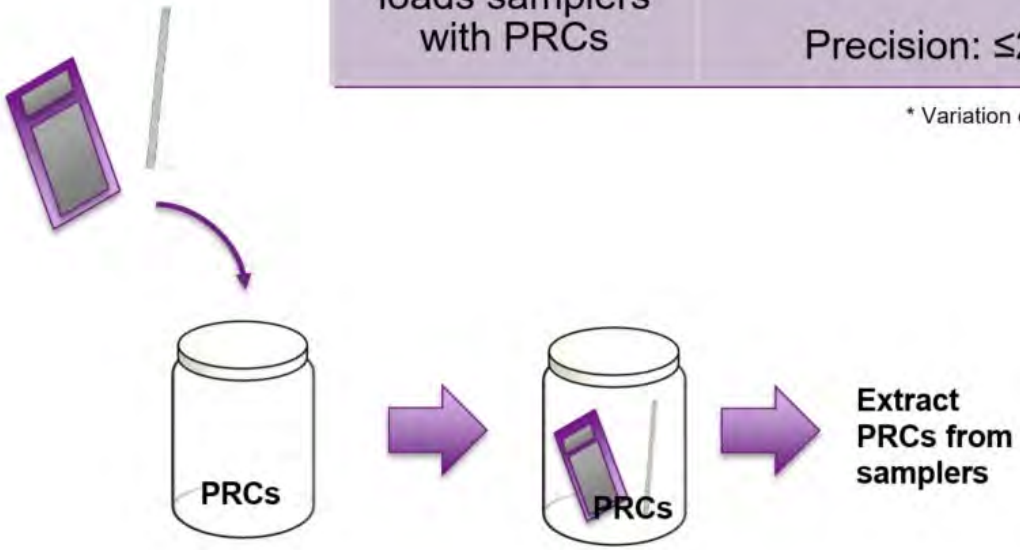

18
72
93
MIT_HighSensitivity
60
Relative Concentration
1.4
1.2
1
0.8
0.6
0.4
0.2
0
13C-PCB 28
13C-PCB 47
13C-PCB 70
13C-PCB 80
13C-PCB 111
13C-PCB 141
13C-PCB 182


86
17
40
MIT_LowSensitivity
Relative Concentration
1.4
1.2
1
0.8
0.6
0.4
0.2
0
13C-PCB 37
13C-PCB 47
13C-PCB 54
13C-PCB 111
13C-PCB 138
13C-PCB 178

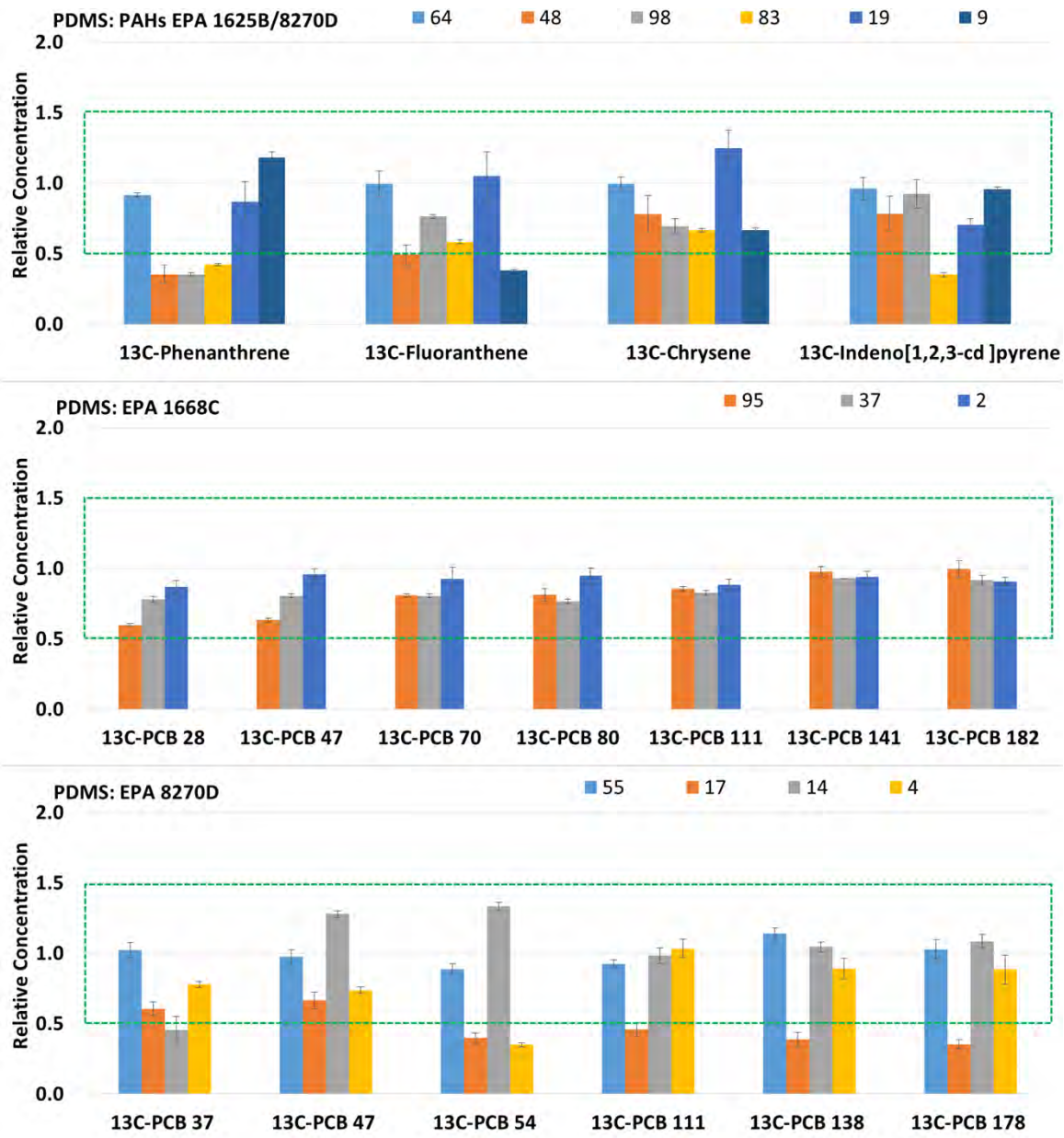
Task 2: Verify PRC Loading Procedures

Each laboratory demonstrates independent start → finish method capability	
Independently loads samplers with PRCs	Accuracy: within ±50% of target concentration achieved* Precision: ≤20% relative standard deviation*

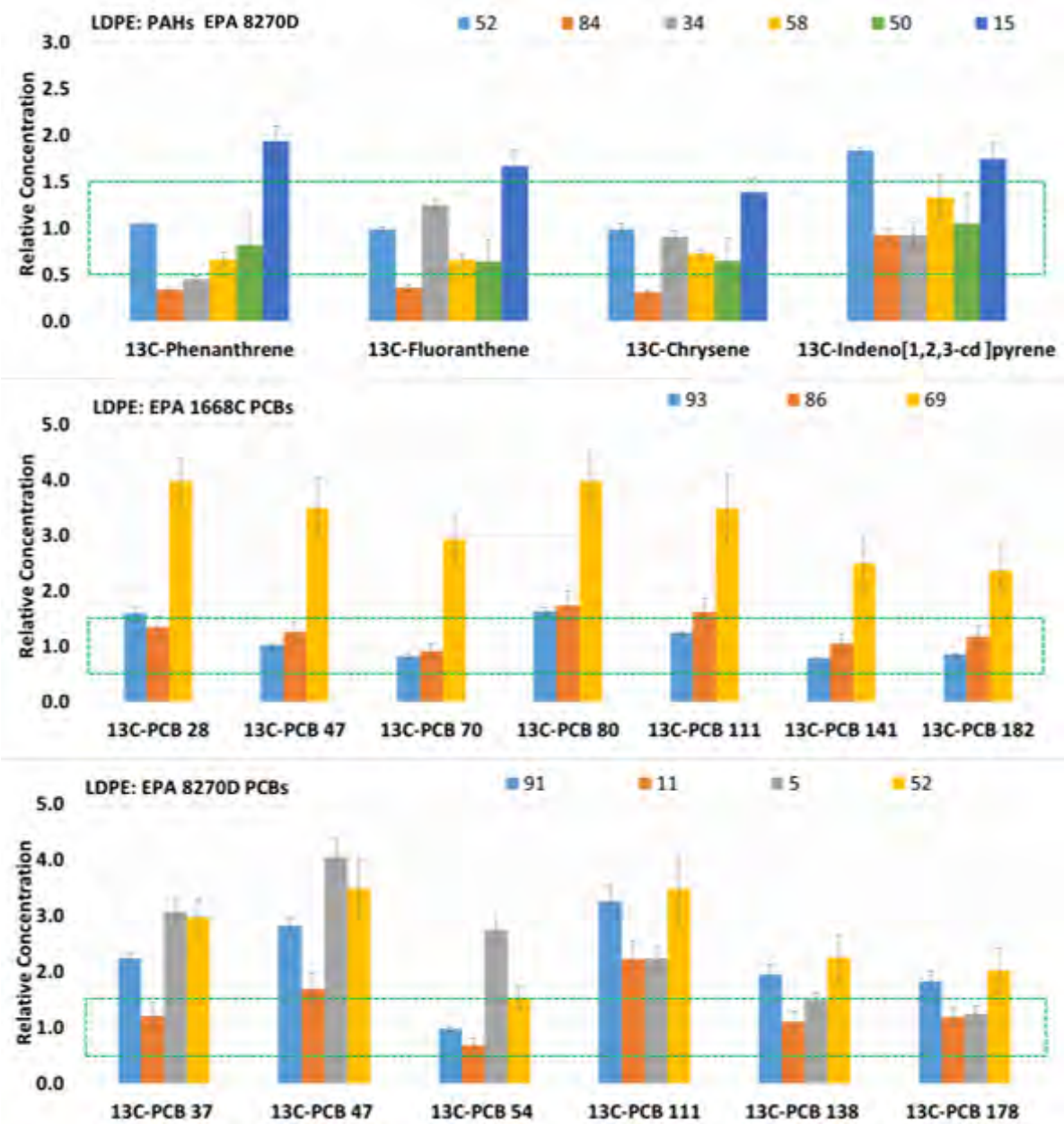
* Variation outside of control limits may be acceptable for one or more targets



PDMS


PDMS: PAHs EPA 1625B/8270D
64
48
98
83
19
9
Relative Concentration
2.0
1.5
1.0
0.5
0.0
13C-Phenanthrene
13C-Fluoranthene
13C-Chrysene
13C-Indeno[1,2,3-cd]pyrene
PDMS: EPA 1668C
95
37
2
Relative Concentration
2.0
1.5
1.0
0.5
0.0
13C-PCB 28
13C-PCB 47
13C-PCB 70
13C-PCB 80
13C-PCB 111
13C-PCB 141
13C-PCB 182
PDMS: EPA 8270D
55
17
14
4
Relative Concentration
2.0
1.5
1.0
0.5
0.0
13C-PCB 37
13C-PCB 47
13C-PCB 54
13C-PCB 111
13C-PCB 138
13C-PCB 178

LDPE

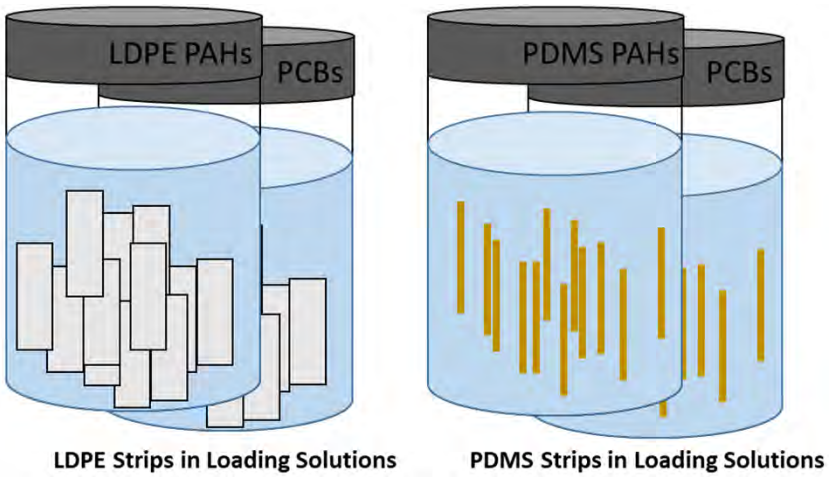

LDPE: PAHs EPA 8270D
52
84
34
58
50
15
Relative Concentration
3.0
2.5
2.0
1.5
1.0
0.5
0.0
13C-Phenanthrene
13C-Fluoranthene
13C-Chrysene
13C-Indeno[1,2,3-cd]pyrene
LDPE: EPA 1668C PCBs
93
86
69
Relative Concentration
5.0
4.0
3.0
2.0
1.0
0.0
13C-PCB 28
13C-PCB 47
13C-PCB 70
13C-PCB 80
13C-PCB 111
13C-PCB 141
13C-PCB 182
LDPE: EPA 8270D PCBs
91
11
5
52
Relative Concentration
5.0
4.0
3.0
2.0
1.0
0.0
13C-PCB 37
13C-PCB 47
13C-PCB 54
13C-PCB 111
13C-PCB 138
13C-PCB 178

Tasks 1 and 2 Conclusions

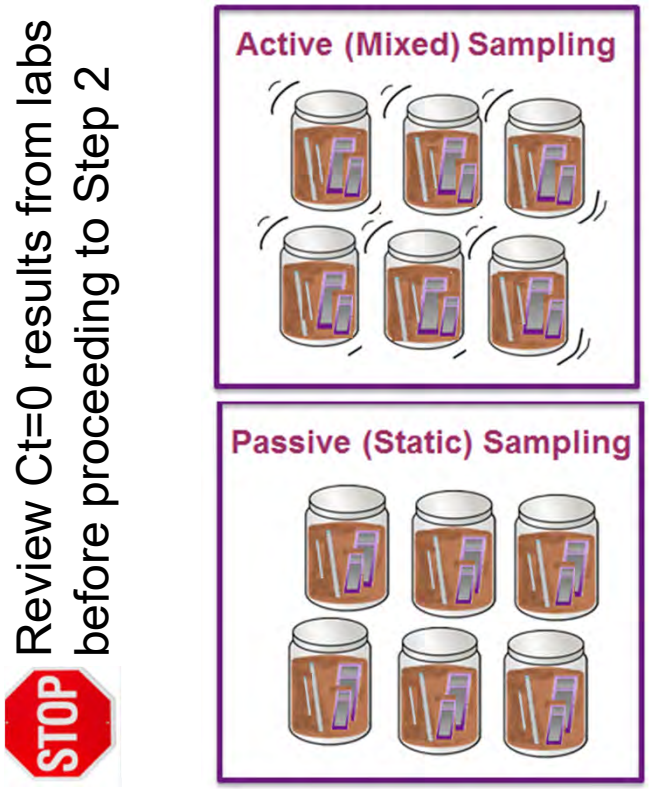
- PAH PRC indeno[1,2,3-cd]pyrene-$^{13}C6$ provides insufficient m/z distinction from column bleed on low resolution GC/MS
- LDPE strip area/weight ratios should be measured and consistent
  - Ensure uniform material prior to PRC loading and sampler use
- Solvent variations can impact final PRC loaded $C_{t=0}$ achieved
  - SOPs were updated to address this issue

Task 3: Inter-Lab Method Validation

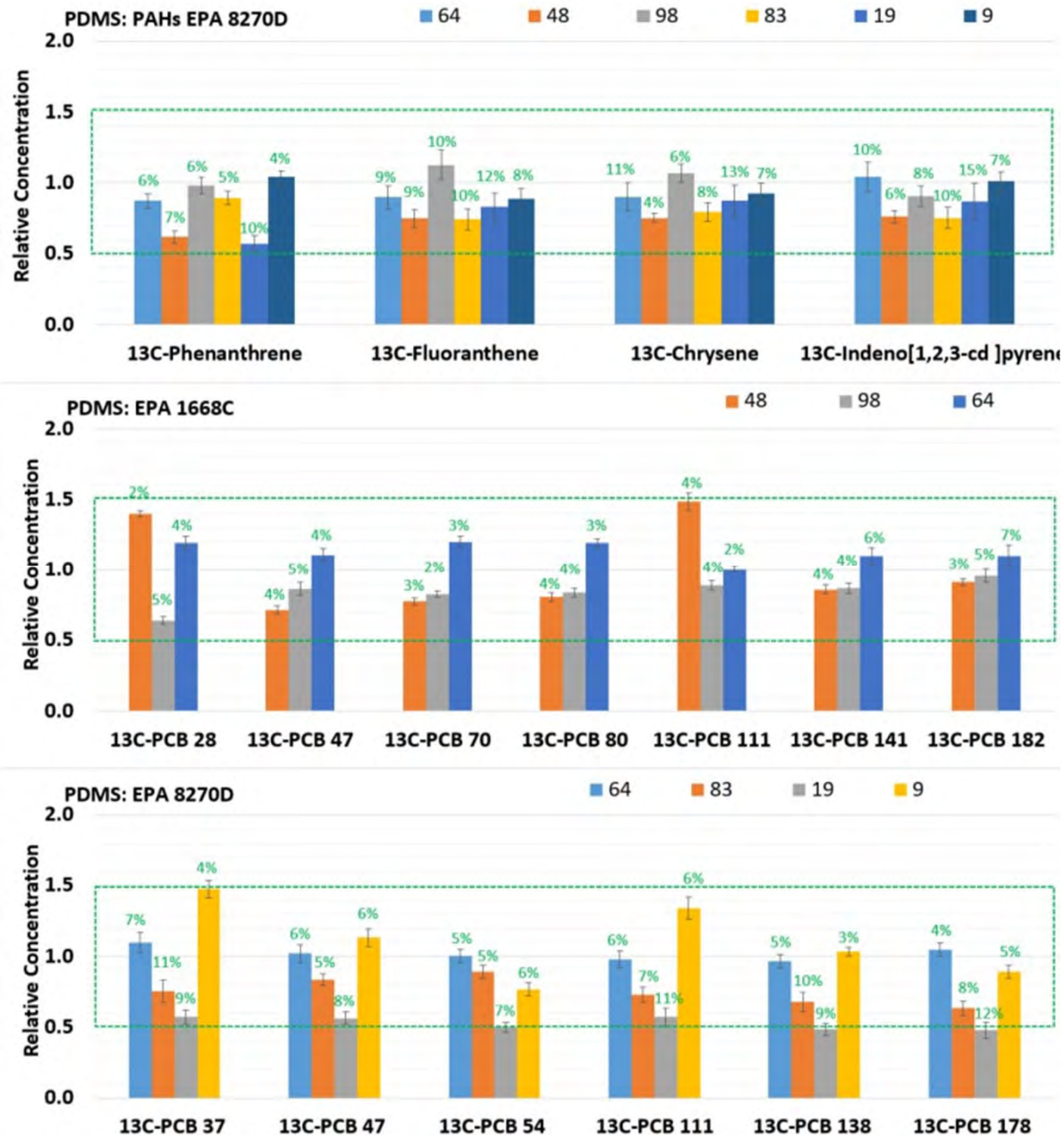
Step 1: PRC Loading



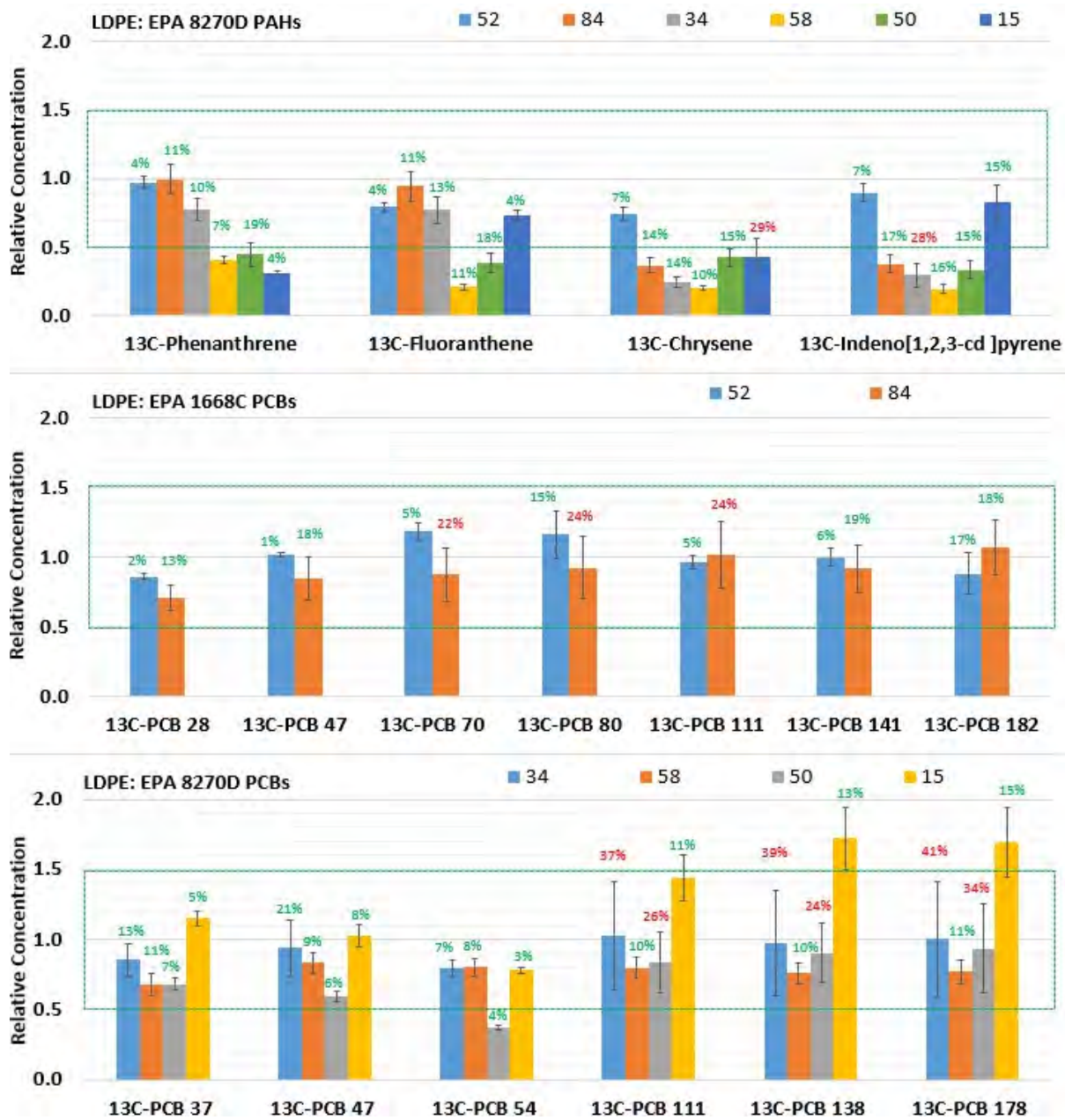
Step 2: Sediment Exposures



PDMS

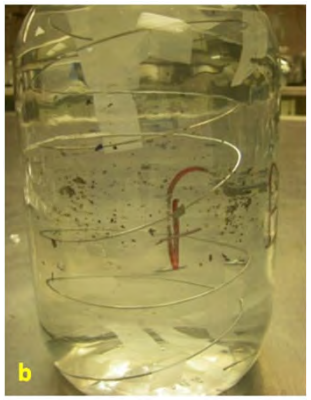

PDMS: PAHs EPA 8270D
64
48
98
83
19
9
Relative Concentration
2.0
1.5
1.0
0.5
0.0
13C-Phenanthrene
13C-Fluoranthene
13C-Chrysene
13C-Indeno[1,2,3-cd]pyrene
PDMS: EPA 1668C
48
98
64
13C-PCB 28
13C-PCB 47
13C-PCB 70
13C-PCB 80
13C-PCB 111
13C-PCB 141
13C-PCB 182
PDMS: EPA 8270D
64
83
19
9
13C-PCB 37
13C-PCB 47
13C-PCB 54
13C-PCB 111
13C-PCB 138
13C-PCB 178

LDPE

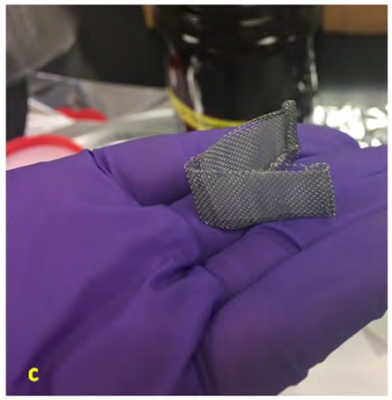

LDPE: EPA 8270D PAHs
52
84
34
58
50
15
Relative Concentration
2.0
1.5
1.0
0.5
0.0
13C-Phenanthrene
13C-Fluoranthene
13C-Chrysene
13C-Indeno[1,2,3-cd]pyrene
LDPE: EPA 1668C PCBs
52
84
13C-PCB 28
13C-PCB 47
13C-PCB 70
13C-PCB 80
13C-PCB 111
13C-PCB 141
13C-PCB 182
LDPE: EPA 8270D PCBs
34
58
50
15
13C-PCB 37
13C-PCB 47
13C-PCB 54
13C-PCB 111
13C-PCB 138
13C-PCB 178

LDPE PRC Loading Troubleshooting

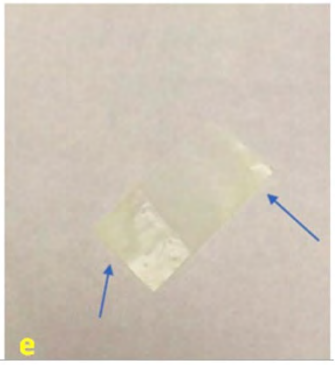
LDPE strips “skewered” along a glass pipet



Strips “skewered” along aluminum coil

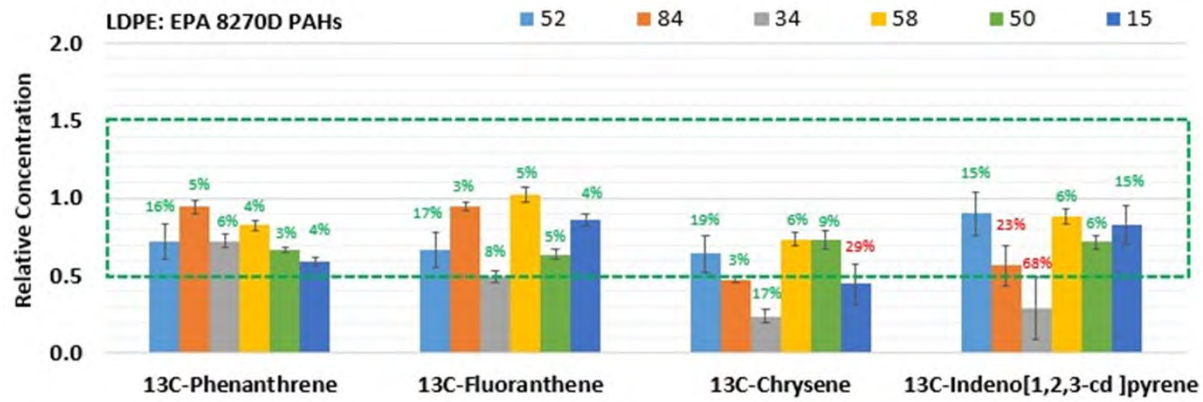


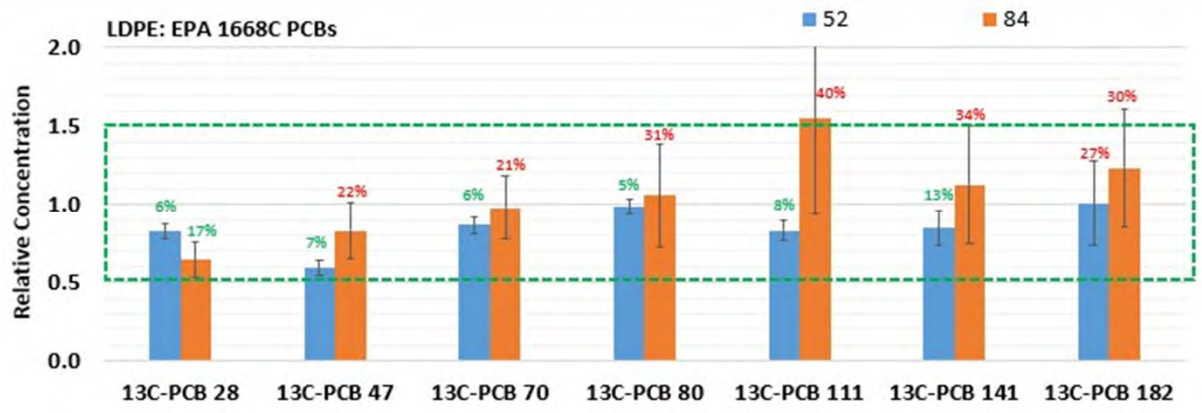
Enclosed in stainless steel mesh

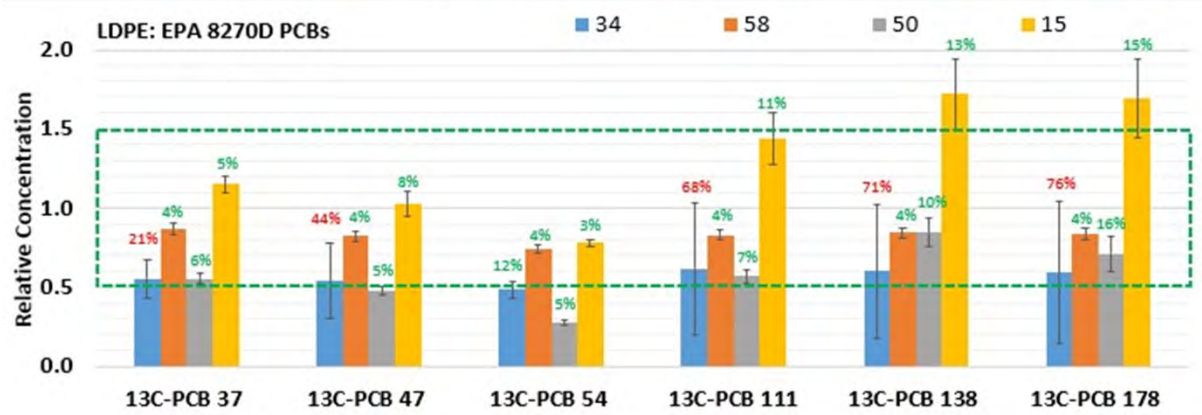


Variation in yellow staining: Uneven loading of PAH PRC indenopyrene?

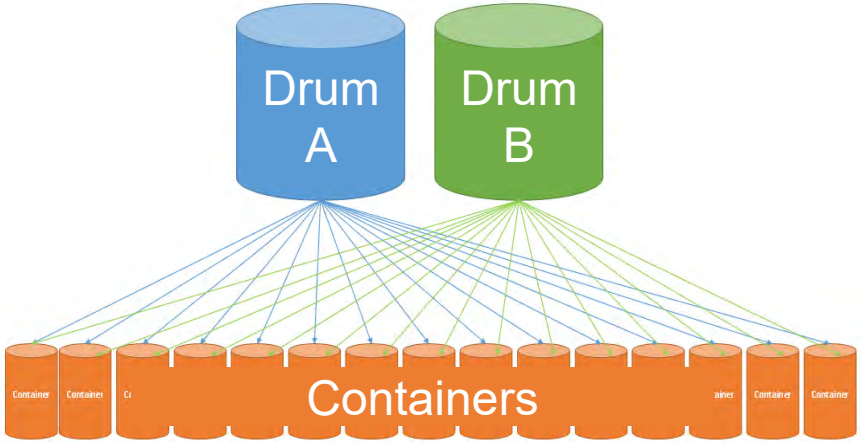
LDPE Final

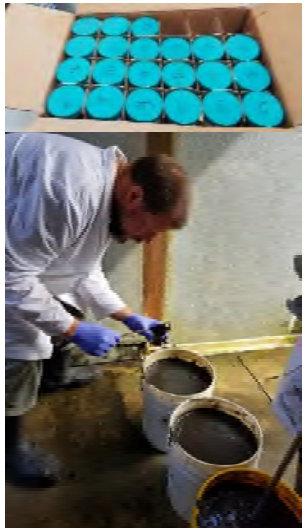

LDPE: EPA 8270D PAHs
52
84
34
58
50
15
Relative Concentration
2.0
1.5
1.0
0.5
0.0
13C-Phenanthrene
13C-Fluoranthene
13C-Chrysene
13C-Indeno[1,2,3-cd]pyrene


LDPE: EPA 1668C PCBs
52
84
Relative Concentration
2.0
1.5
1.0
0.5
0.0
13C-PCB 28
13C-PCB 47
13C-PCB 70
13C-PCB 80
13C-PCB 111
13C-PCB 141
13C-PCB 182


LDPE: EPA 8270D PCBs
34
58
50
15
Relative Concentration
2.0
1.5
1.0
0.5
0.0
13C-PCB 37
13C-PCB 47
13C-PCB 54
13C-PCB 111
13C-PCB 138
13C-PCB 178

Task 3 Sediment Characterization





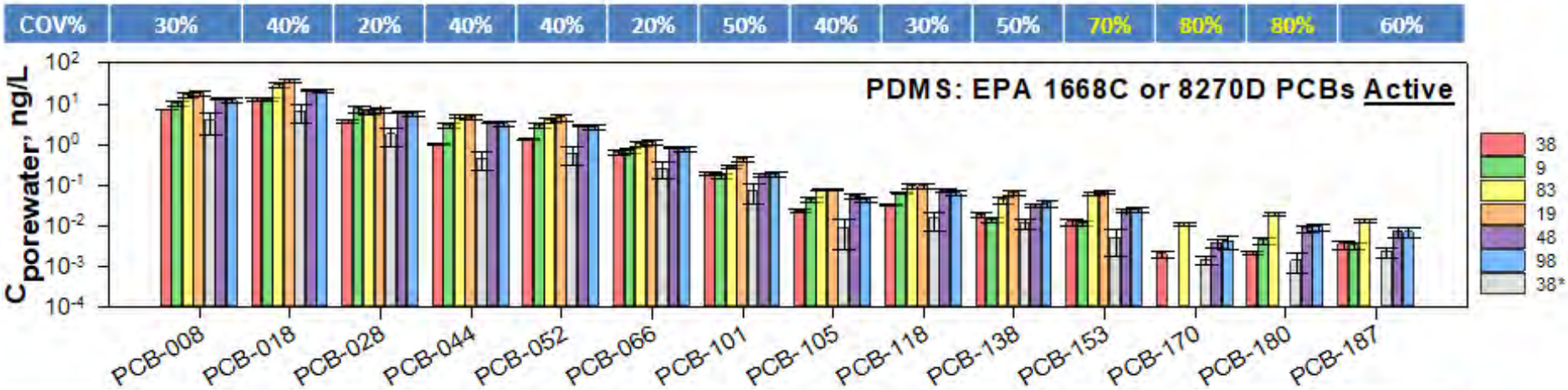
Sediment Characterization Results

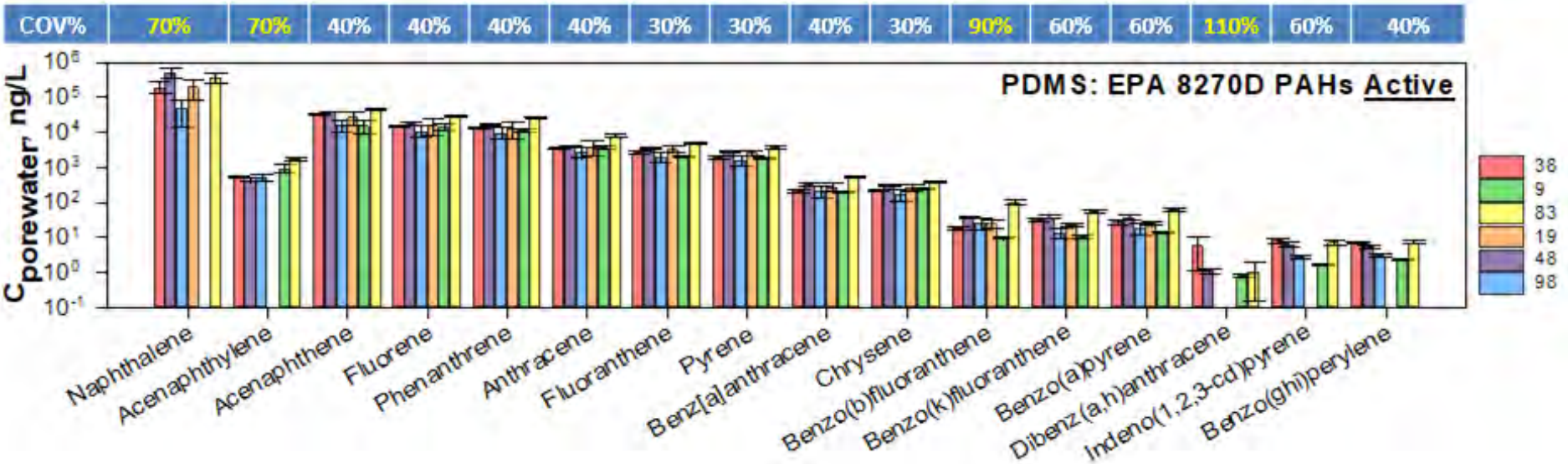
PAHs	Average, ug/kg dry weight	Standard Deviation	Relative Standard Deviation
Naphthalene	415,750	64,385	15%
Phenanthrene	29,500	4,241	14%
Anthracene	55,075	3,164	6%
Fluoranthene	20,483	3,090	15%
Pyrene	17,575	2,615	15%
Benzo (a) anthracene	44,267	2,963	7%
Chrysene	57,058	4,023	7%
Benzo (b) fluoranthene	32,833	2,186	7%
Benzo (k) fluoranthene	48,017	6,006	13%
Benzo (a) pyrene	48,275	3,195	7%
Indeno (1,2,3-cd) pyrene	20,533	2,110	10%
Dibenz (a,h) anthracene	6,465	902	14%
Benzo (g,h,i) perylene	22,625	2,492	11%
Acenaphthylene	3,724	369	10%
Acenaphthene	110,508	25,743	23%
Fluorene	67,550	21,170	31%
Sum, mg/kg dw	1,000		

PCBs	Average, ug/kg dry weight	Standard Deviation	Relative Standard Deviation
PCB 8	327	19	6%
PCB 101	123	8	7%
PCB 105	50	3	7%
PCB 118	73	6	8%
PCB 126	ND	--	--
PCB 128	ND	--	--
PCB 18	689	63	9%
PCB 138	52	4	8%
PCB 153	54	5	9%
PCB 28	951	69	7%
PCB 169	ND	--	--
PCB 170	19	2	11%
PCB 180	44	4	8%
PCB 187	22	2	9%
PCB 44	309	21	7%
PCB 52	275	21	8%
PCB 66	270	21	8%
PCB 77	47	4	9%
Sum, mg/kg dw	3.3		

Inter-Lab Method Validation Results

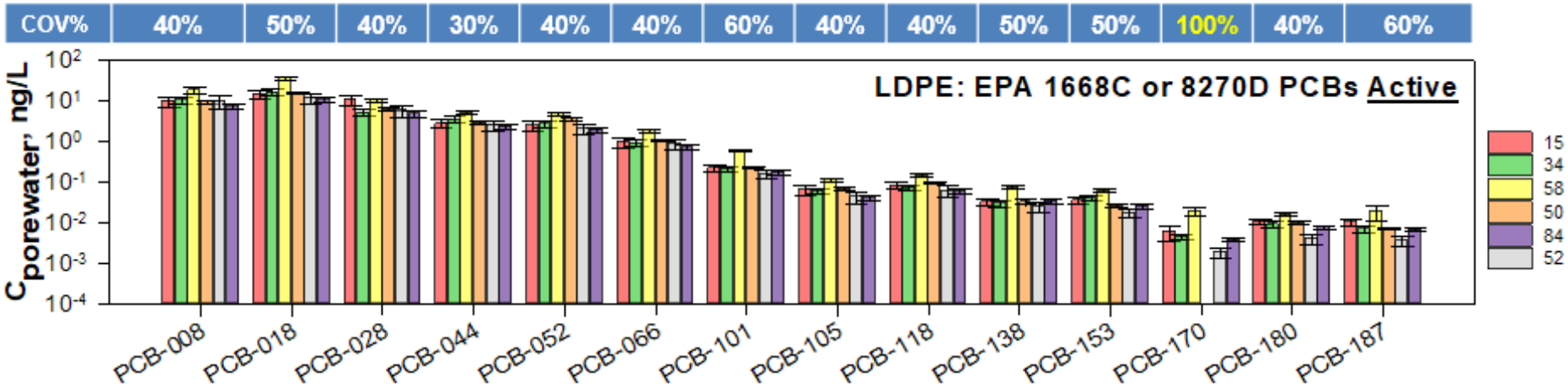
PDMS "Active" Exposures

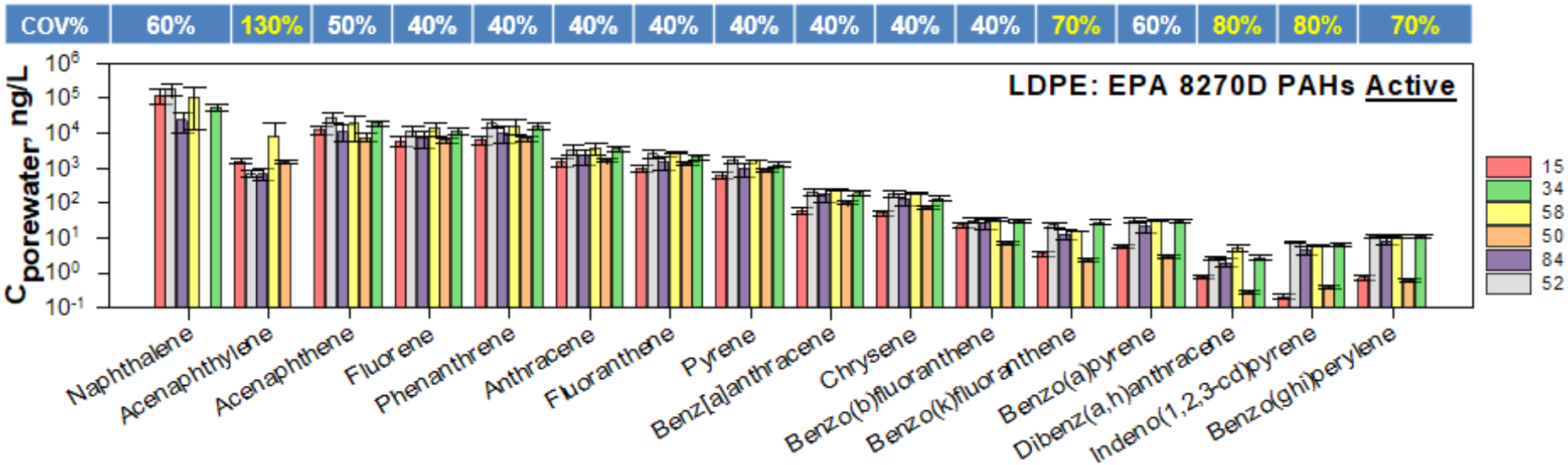




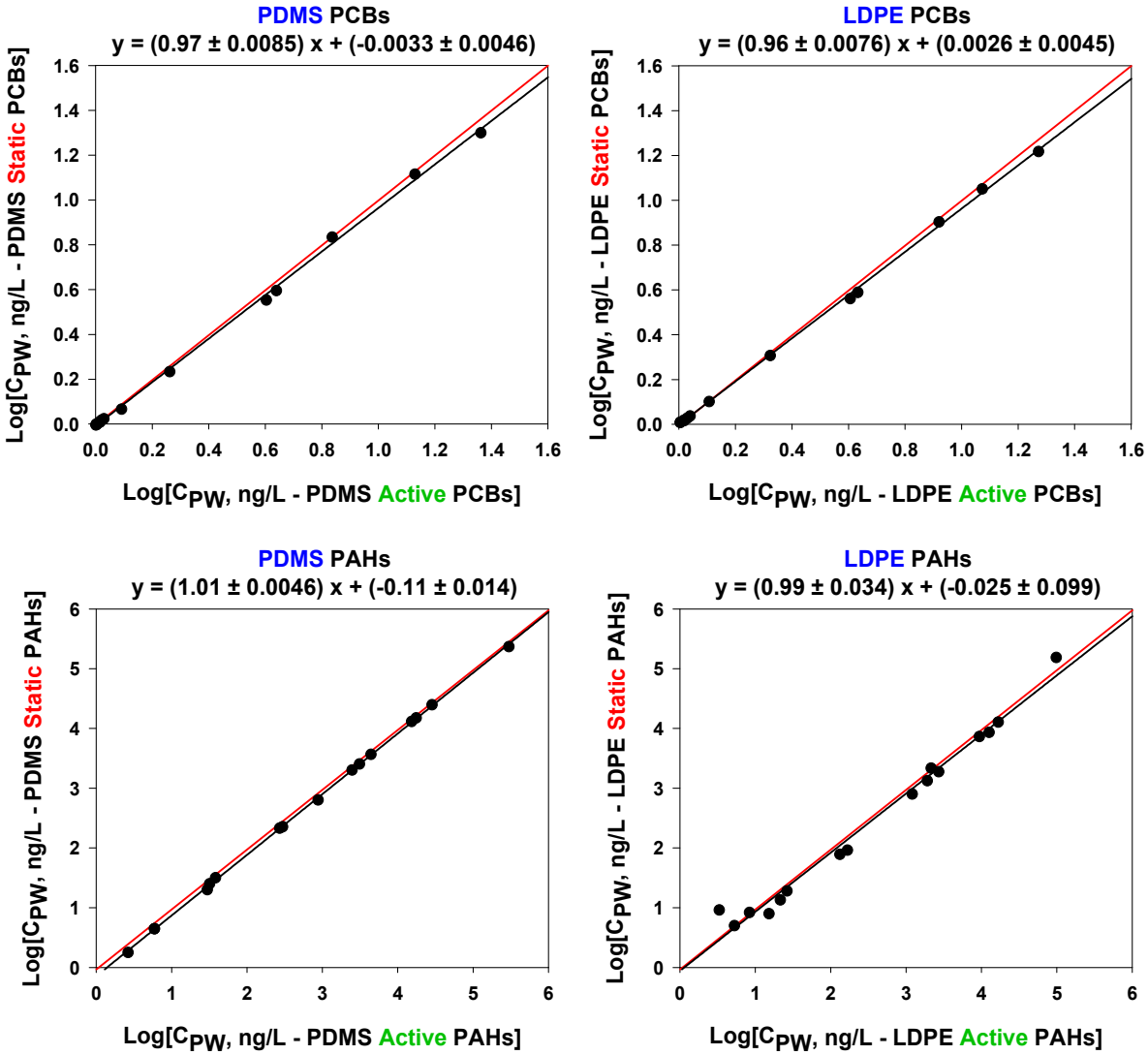
Inter-Lab Method Validation Results

LDPE "Active" Exposures



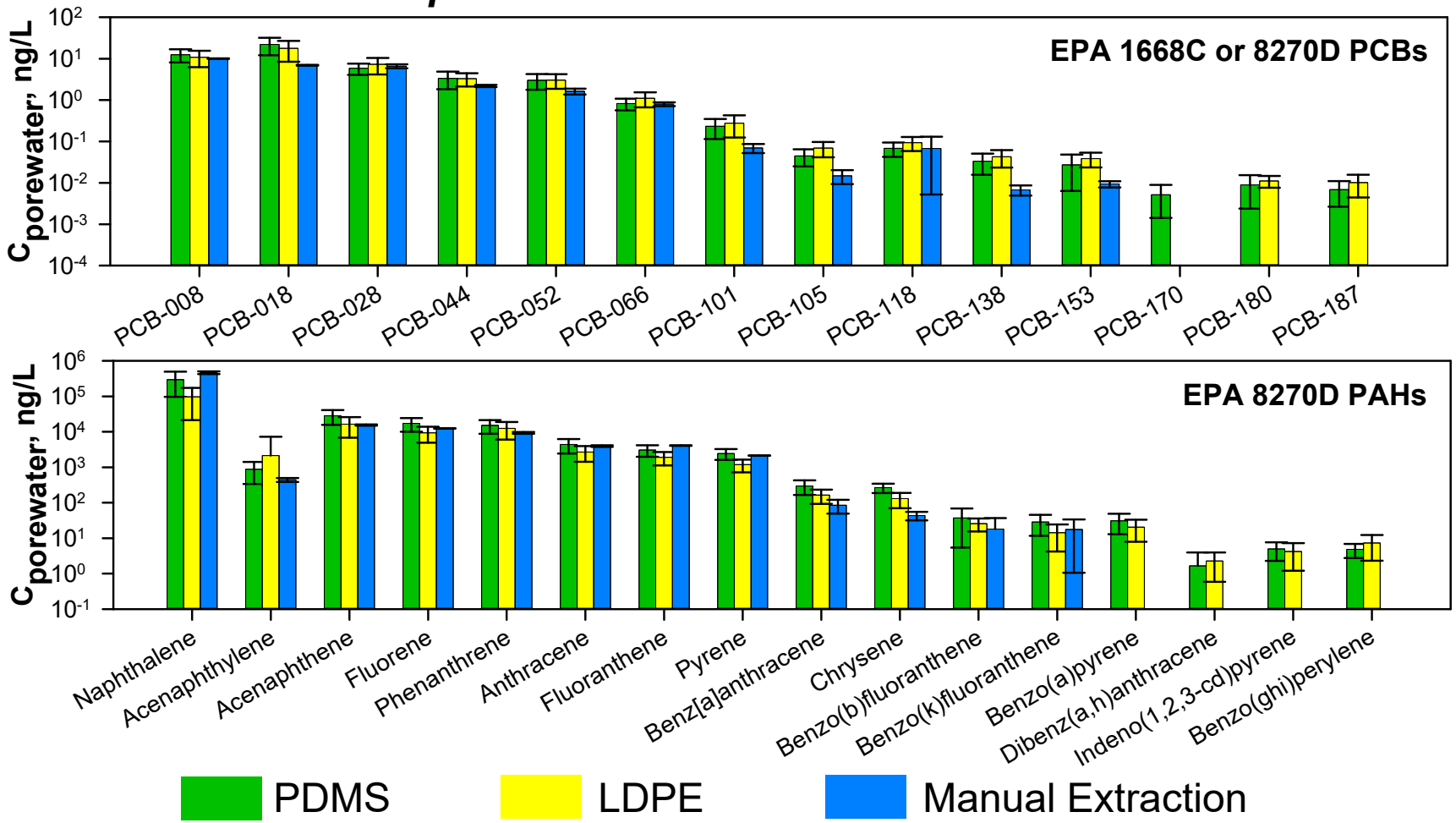


Active vs. Static Exposures



Comparison of C*free* Results

Polymeric sampler (LDPE & PDMS) vs. manual porewater extraction reference method



Conclusions

Key study objectives achieved!

- Four commercial labs
  - Meets SW-846 method application requirement
- Improved final SOPs
  - Phased approach with troubleshooting
- Increased commercial availability
- Accelerated technology transfer
  - Instructional videos, webinars, peer-reviewed manuscript in-progress

Good agreement between labs and methods

- Static exposures in agreement with well-mixed active exposures
  - PRCs recommended for in situ field sampling
  - Optional for active lab exposures
- Polymeric sampler measurements
  - In agreement with manual porewater extraction measurements for compounds also measured using that method

Benefits to DoD

Increased Commercial Availability

BATTELLE
It can be done

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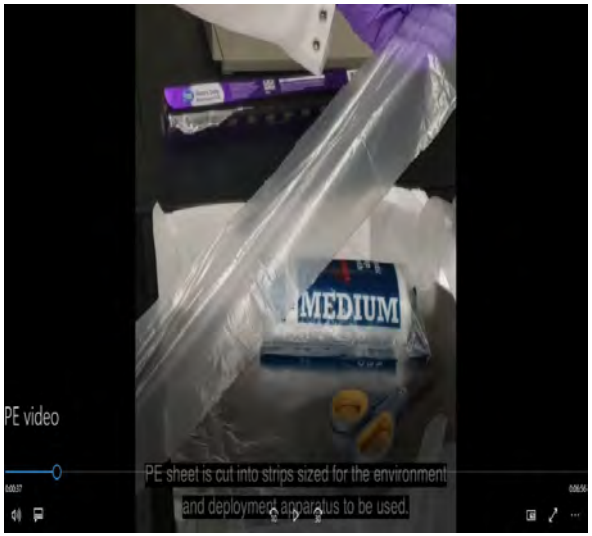
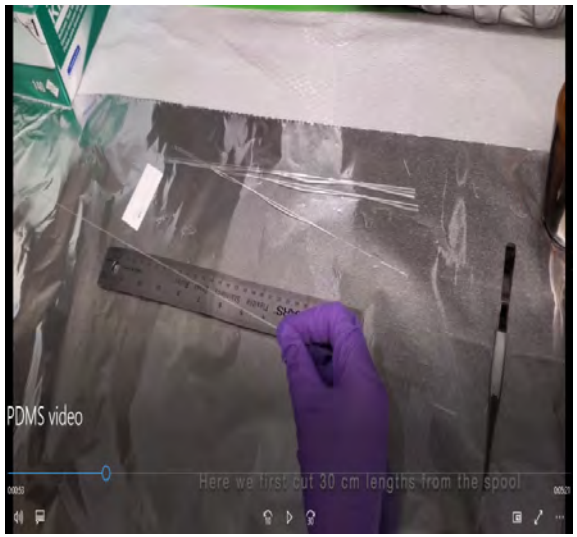
Benefits to DoD

Technology Transfer – Promotion of the Standardized Method





- Polyethylene
  - https://www.youtube.com/watch?v=XjE4ZoCQIPk&feature=youtu.be&ab_channel=Passivesampling
- PDMS
  - https://www.youtube.com/watch?v=d0k4JWKm_Ls&feature=youtu.be&ab_channel=Passivesampling

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- Phil Gschwend, John MacFarlane
  - Massachusetts Institute of Technology
- Rob Burgess, Matt Lambert, Marc Mills
  - U.S. EPA
- Mingta Lin
  - Pyron Environmental

For additional information, please visit

https://www.serdp-estcp.org/Program-Areas/Environmental-Restoration/Contaminated-Sediments/Bioavailability/ER-201735

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Q&A Session 1

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Long Term Monitoring of Activated Carbon Amendment in Sediments at an Active Shipyard

Gunther Rosen
Naval Information Warfare Center Pacific

SERDP DOD • EPA • DOE | ESTCP

Agenda

- Demonstration objectives
- Amendment and demonstration site description
- Placement considerations and verification
- Long-term monitoring design
- Long-term monitoring results
- Key points and considerations

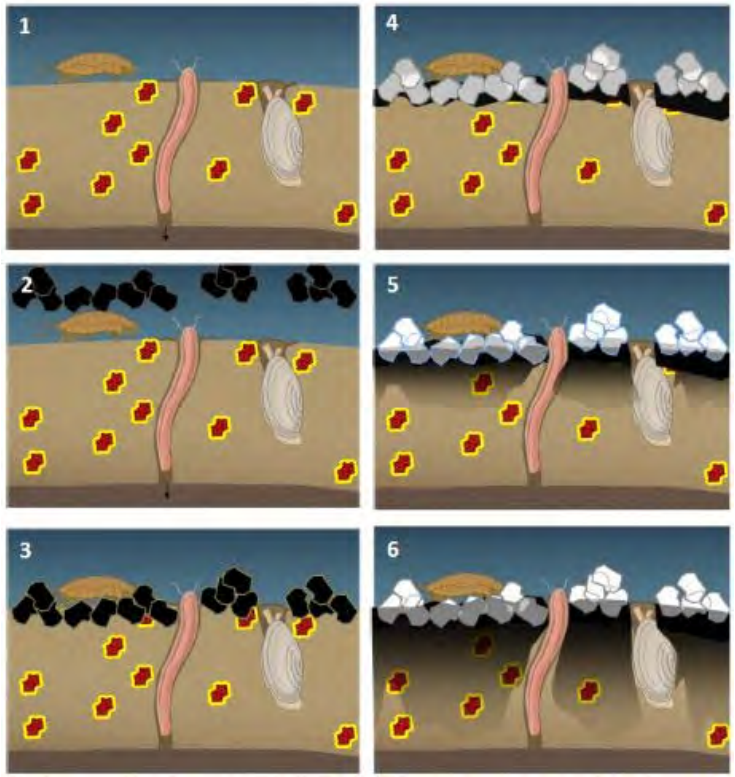
Demonstration Objectives

- Demonstrate long-term stability and performance of an AC amendment for treatment of contaminated sediments in an active DoD harbor in deeper water areas
- Add-on effort to ESTCP Environmental Restoration Project of the Year (2016)
- Long term focus (~7 years post-placement)
  - Effectiveness of amendment in controlling contaminant bioavailability
  - Physical stability and longevity of the amendment in the sediment
  - Response of the benthic community to the site amendment application

Sources: *ESTCP ER-201131; Kirtay et al. 2018. Environ. Toxicol. Chem , 37, 1767-1777*

AquaGate Reactive Amendment

- Cost-effective alternative to dredging
- AquaGate+PAC™
  - AquaBlok®, Toledo, Ohio
- Coated aggregate
  - Coating: 5% Powder Activated Carbon (PAC) with ~10% bentonite binder
  - Aggregate aids delivery of PAC to sediment surface
- Penetrates into sediment due to aggregate settling
- Vertically mixed by natural processes over time



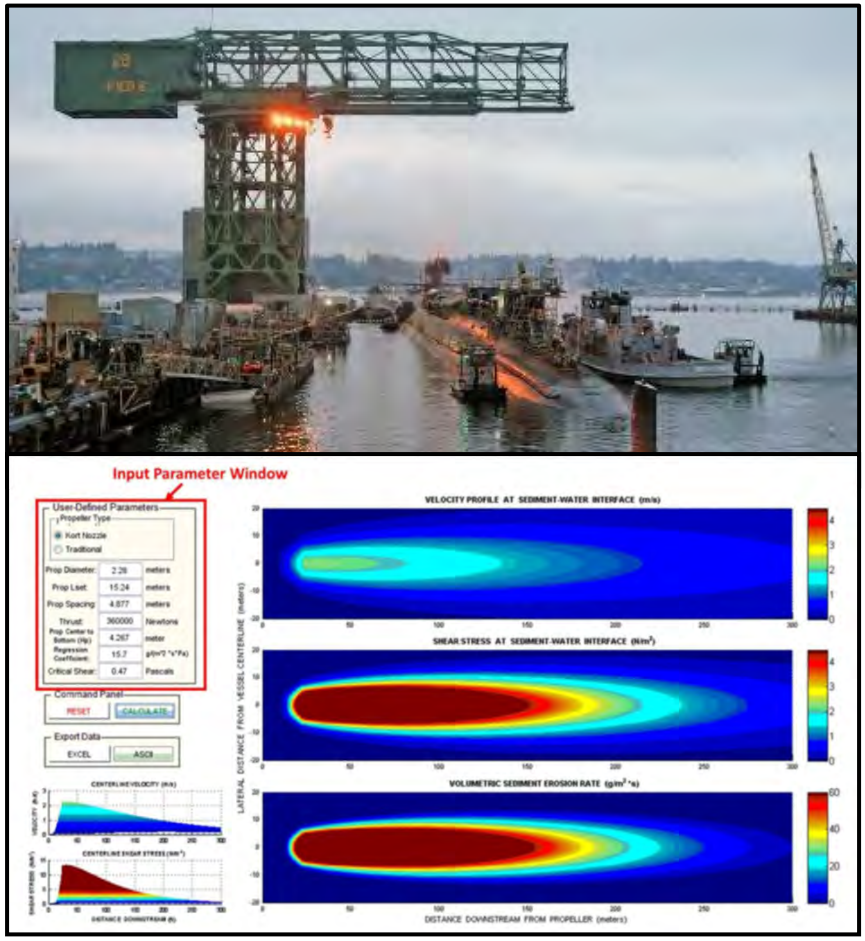
Demonstration Site

Pier 7, Puget Sound Naval Shipyard, Bremerton, WA

- Active, deep water shipyard
- Operable Unit B Marine
- Highest PCB concentrations in surface sediment
  - Along the shoreline
  - Adjacent to moorings and piers
  - Previously dredged
- Continued presence of elevated PCBs near Pier 7
- Desire to test alternative in situ treatment methods
  - Reactive amendments

Potential Physical Disturbance

- Believed to be primarily driven by ship movements
- ~4 events/year at Pier 7
  - Each erodes ~20,000 kg sediment
- Scour area
  - 10-20 m wide
  - 50-150 m long
  - 1-5 cm deep



Sources: *ESTCP ER-201031; ESTCP ER-201131*

Amendment Placement

2012 Amendment Placement Verification

- 0.5-month monitoring event
  - No ship movements
  - Sediment Profile Imaging (SPI)[1]
  - ~80% of target area received at least a trace of amendment
  - ~70% of the target area received target thickness (5 cm) or more
- Thickness within target amendment area
  - Average 11 cm
  - Range 0.1 – 17 cm

Average AC Layer Thickness (cm)
Red line = target amendment area

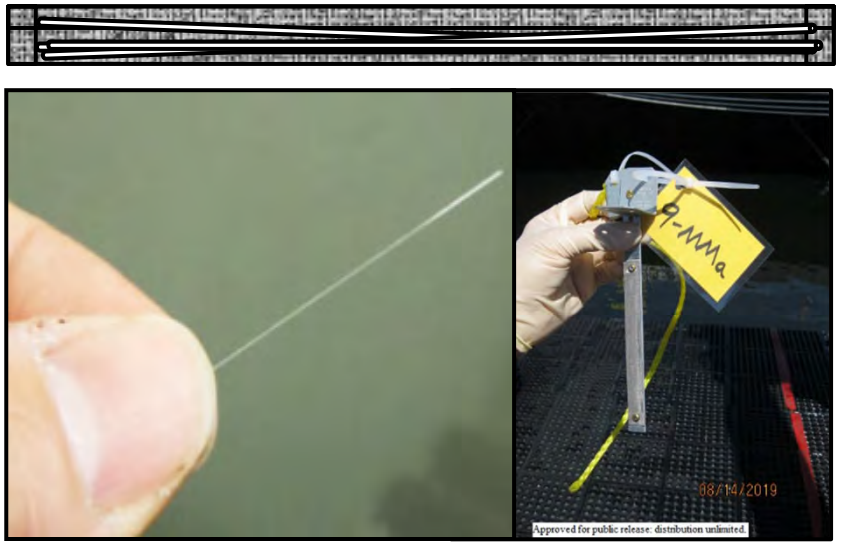
***Source**: [1]Germano and Associates*

Demonstration Design

PCB Availability

- In situ bioaccumulation
  - 10 stations with SEA Rings[1]
  - *Macoma nasuta* (bent-nosed clam)
  - *Nephtys caecoides* (polychaete)

- In situ porewater passive sampling
  - SPME
  - 10 fibers w/ PDMS coating
  - Loaded with 8 PRCs
  - Stainless steel envelope w/ bracket for stability



***Note**: PDMS = polydimethylsiloxane; SPME = solid-phase microextraction; [1]ESTCP ER-201130*

Demonstration Design

Activated Carbon (AC) Persistence Assessed Laterally and with Depth

- Collection and sectioning of multiple cores adjacent to SEA Ring
- Total Organic Carbon (TOC) Lloyd Kahn
- Black Carbon (BC)
  - Chemical oxidation[1]
    - Acid oxidation of NOM while preserving activated carbon (AC)
  - Carbon petrography[2]
    - Visual, photomicroscopic evaluation of sediment
    - AC visually distinct from other components
    - AC can be quantified (cross-sectional volume) and used to estimate AC content (% w/w)

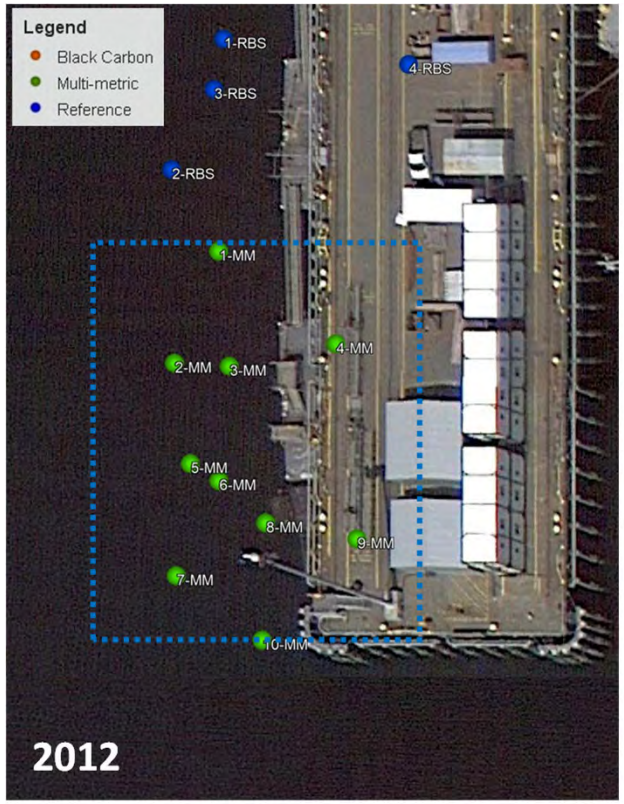
Sources: *[1]Ghosh and Grossman, Chemosphere 2009, 75, 469-475; [2]Ghosh et al., Environ. Sci. Technol. 2003, 37, 2209-2217*

Demonstration Design

Supporting Measurements

- Benthic community assessment to evaluate potential adverse affects of AC
  - 6 benthic metrics
- Sediment bulk chemistry
- Evaluation of core samples for aggregate presence
  - Potential indicator of persistence

Demonstration Site and Sampling Locations



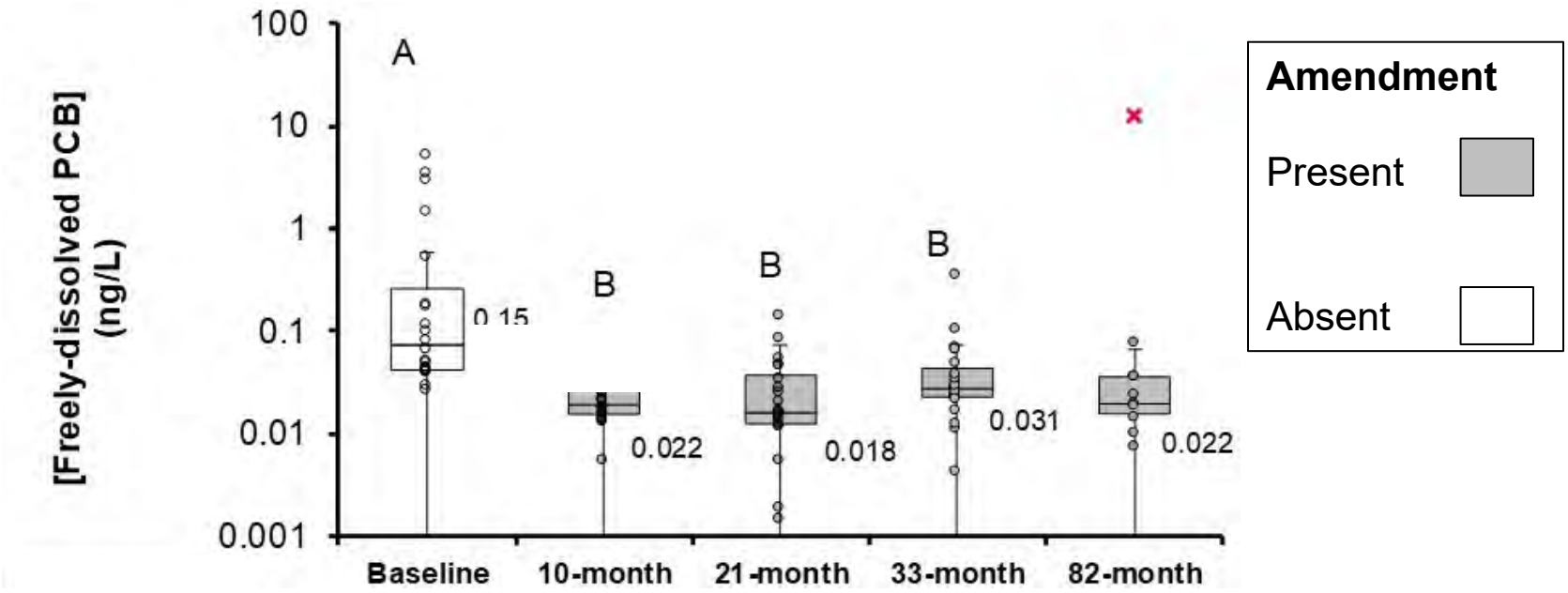
Amended area

Additional stations for enhanced black carbon (2019)

Multi-metric stations (2012-2019)

Benthic reference locations (2012-2019)

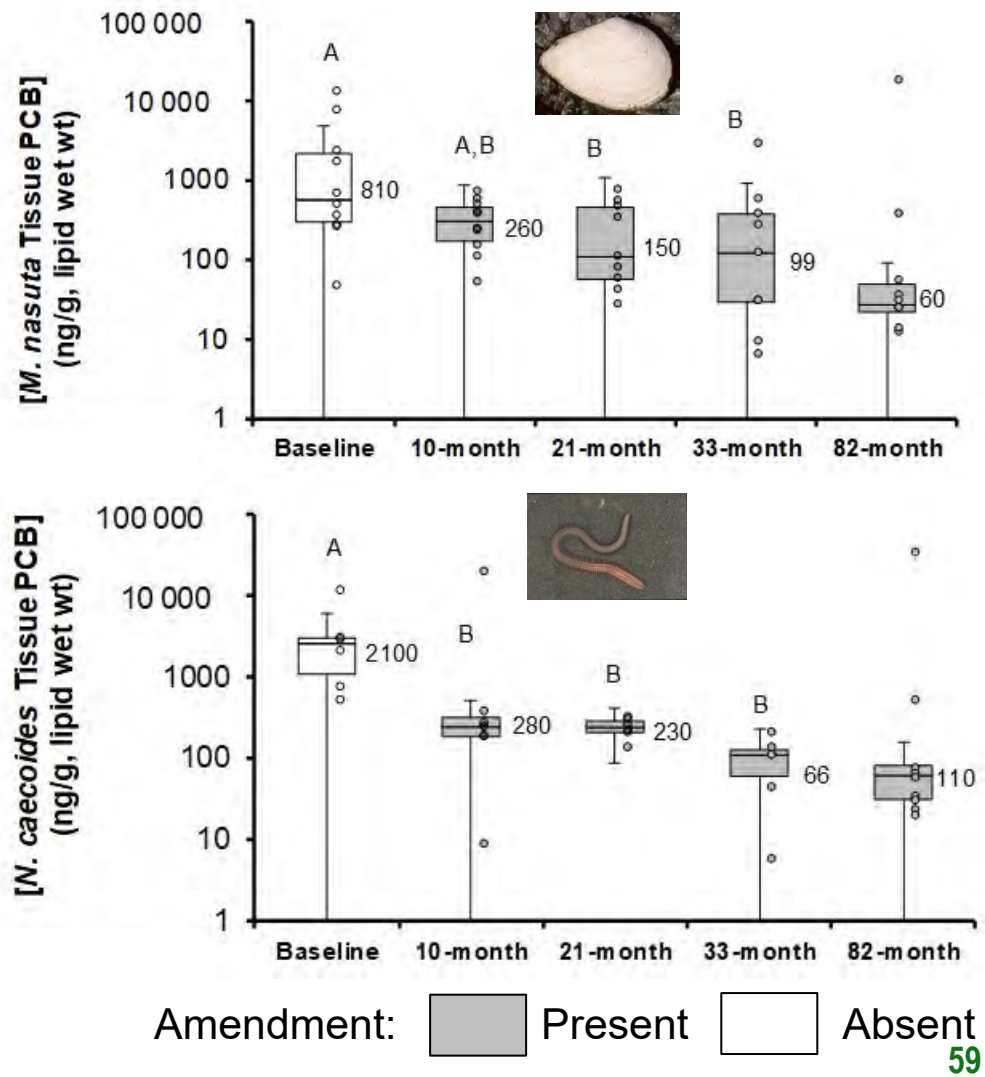
Porewater PCB Reduction (SPME)



- PCBs at 82 months low
  - Consistent with the earlier post-amendment results
- 80-90% lower than baseline
- Large number of NDs at 82 months
  - Used ½ MDL for NDs for all time points

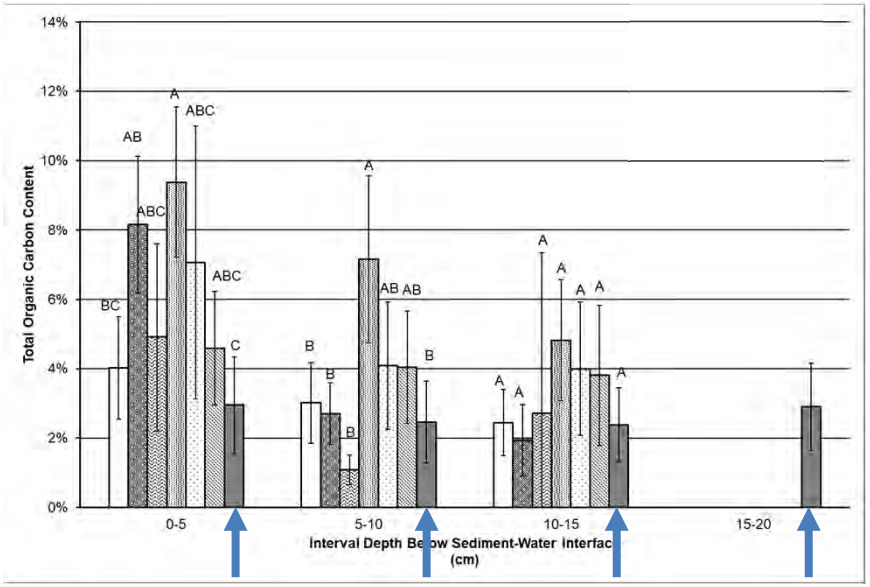
Tissue PCB Reduction

- PCBs at 82 months low
  - Consistent with previous post-amendment monitoring results
- AC amendment is continuing to keep PCB availability ~80-90% < baseline
- Large number of ND at T=82 mo → used ½ MDL for NDs for all time points

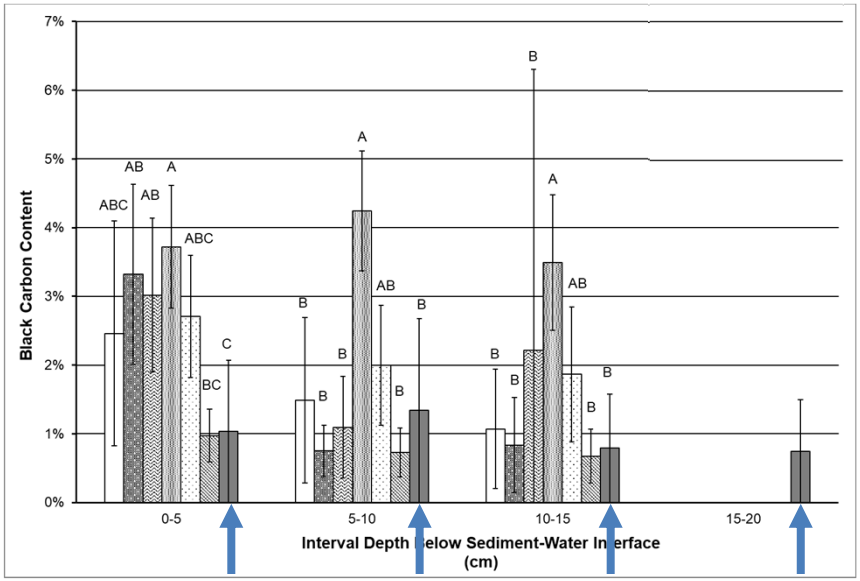


TOC and BC in Sediment: 82 Months

TOC



BC



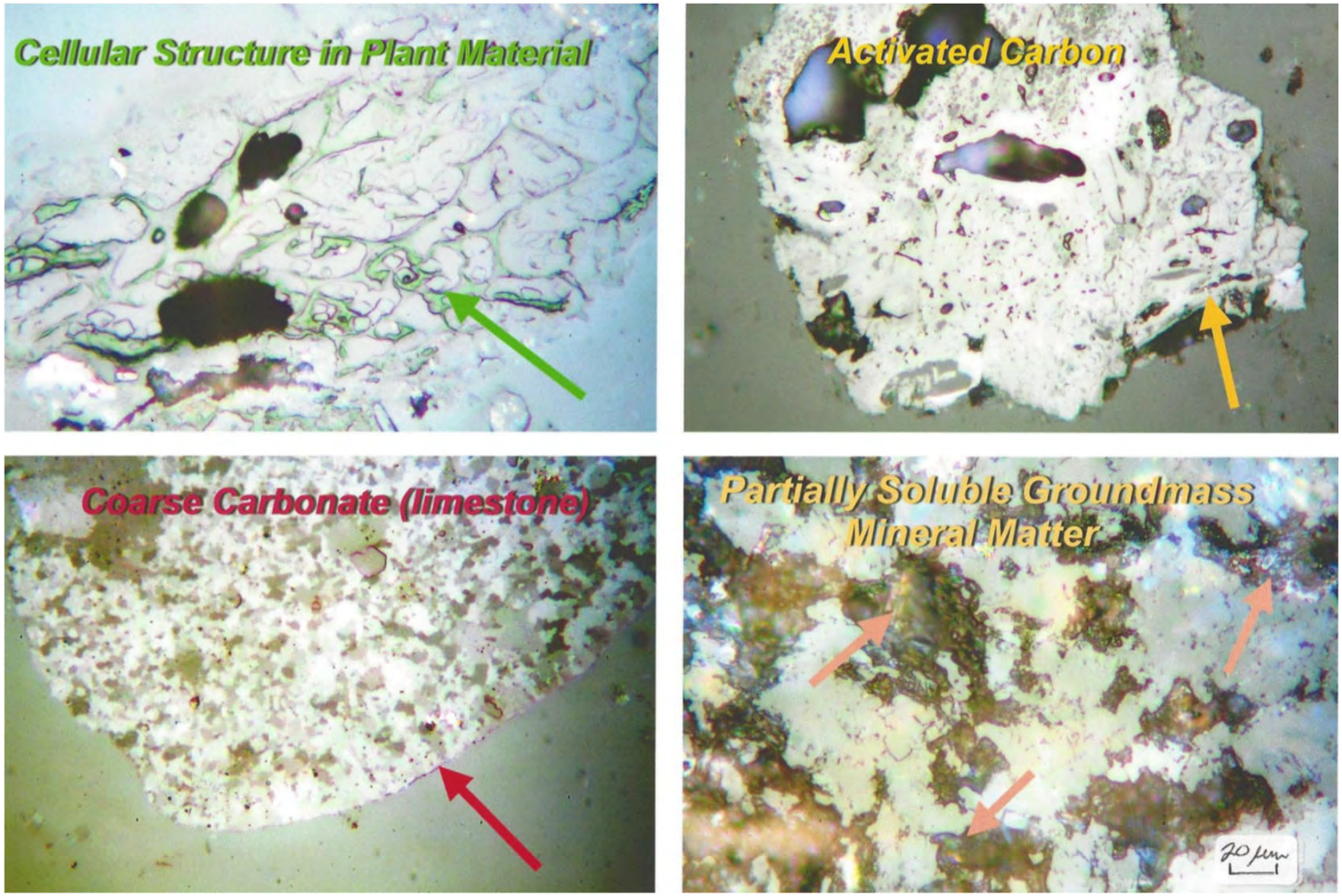
Baseline | 3-Month | 21-Month | 82-Month

0.5-Month | 10-Month | 33-Month

TOC and BC in Sediment: 82 Months

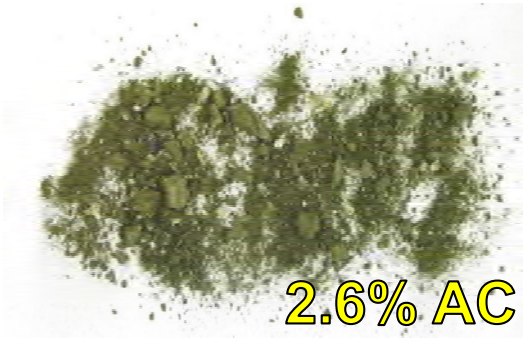
- Stable, comparable to levels observed in 33-month event
- TOC: 2-2.5% on average
- BC: 0.8-1.3% on average
  - Indicates continued presence of AC in the top 20 cm of sediment
- No statistically-significant differences among the 4 layers
  - 0-5, 5-10, 10-15, 15-20 cm

Carbon Petrography



Materials identified in Pier 7 sediment cores from 82-month sampling event; 600X magnification

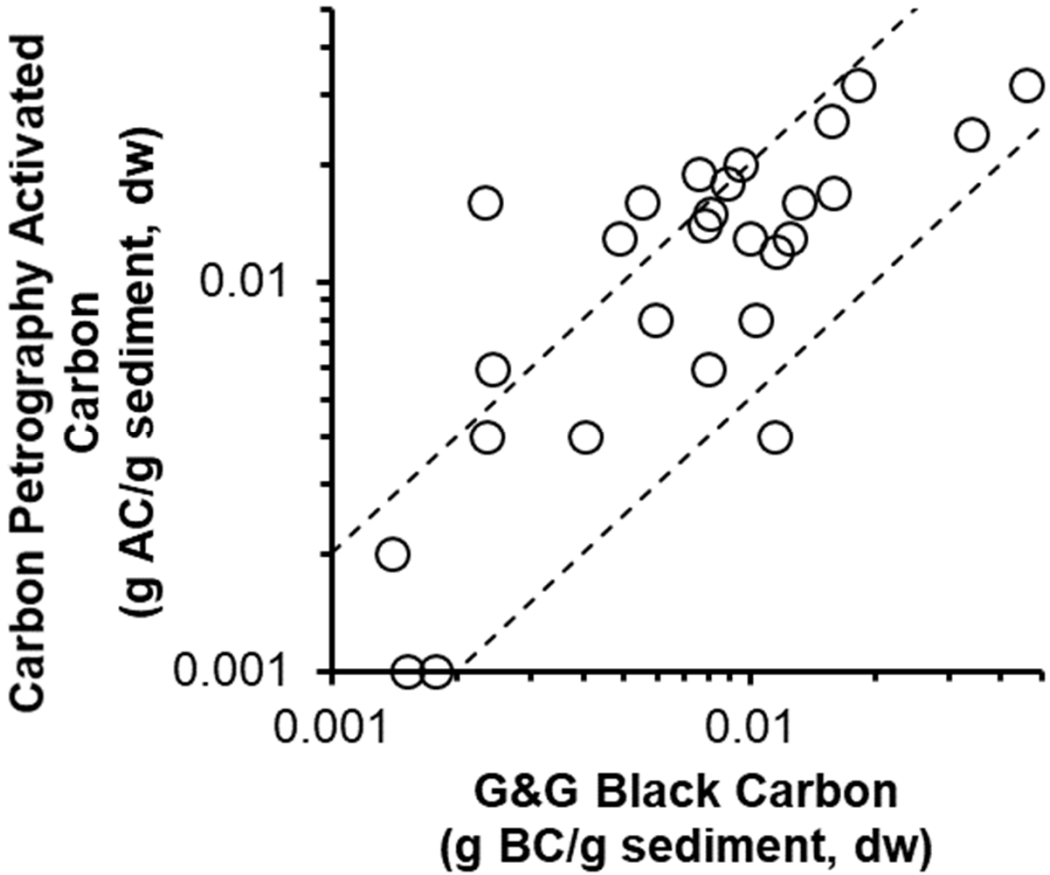
Select Dried Examples Prior to Petrographic (Microscopic) Analysis



Sample Type	Sample ID	BC (% by wt)	AC (% by vol)
Low carbon	2020-0081/T82-3-MM-BC-0005	0.1%	0.2%
Medium carbon	2020-0704/T82-4-MM-BC-0510	1.6%	2.6%
High carbon	2020-0698/T82-2-MM-BC-051	4.6%	3.2%

Presence of aggregate at same size of AquaGate+PAC a potential indicator of AC presence

Comparison of Petrography and BC Results



Note: Symbols between the dashed lines indicate a factor of 2 agreement (or better) between AC and BC

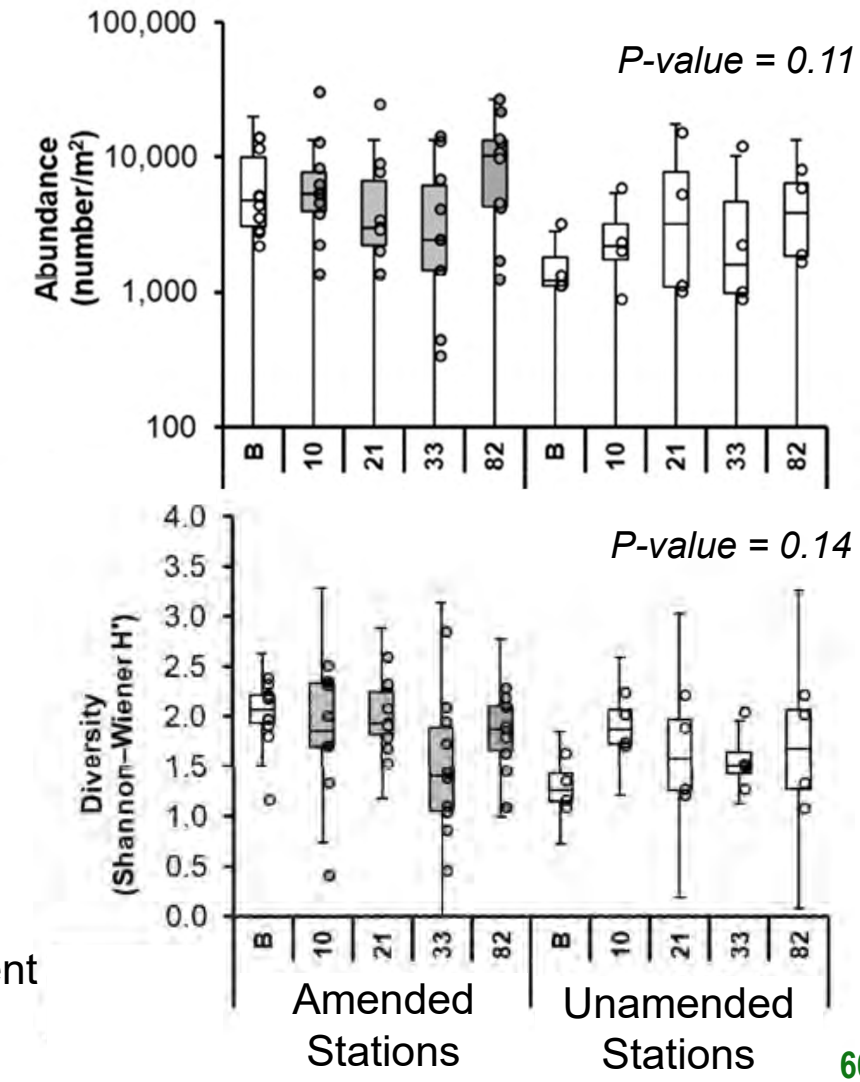
TOC, BC, and AC – 82-Month Data

Aggregate Present	TOC (EPA Lloyd Khan Method)			BC (Grossman and Ghosh 2009)			AC (Carbon Petrography)		
	n	Avg	SD	n	Avg	SD	n	Avg	SD
W/ Aggregate	56	2.9%	1.7%	58	1.3%	1.2%	26	1.3%	0.9%
No Aggregate	32	2.6%	1.4%	30	0.3%	0.2%	6	0.3%	0.2%
Overall		**2.7%**			**1.0%**			**1.1%**	

- 23 locations cored
  - 20 (87%) had aggregate in at least one layer
- Samples w/ aggregate
  - ~4X higher BC or AC content than no aggregate
- If BC is highly present at site, could characterize the site using Petrography at baseline prior to AC treatment
  - Grossman and Ghosh (2009) BC method seems sufficient in this case

Potential for Adverse Impact to Native Benthic Community

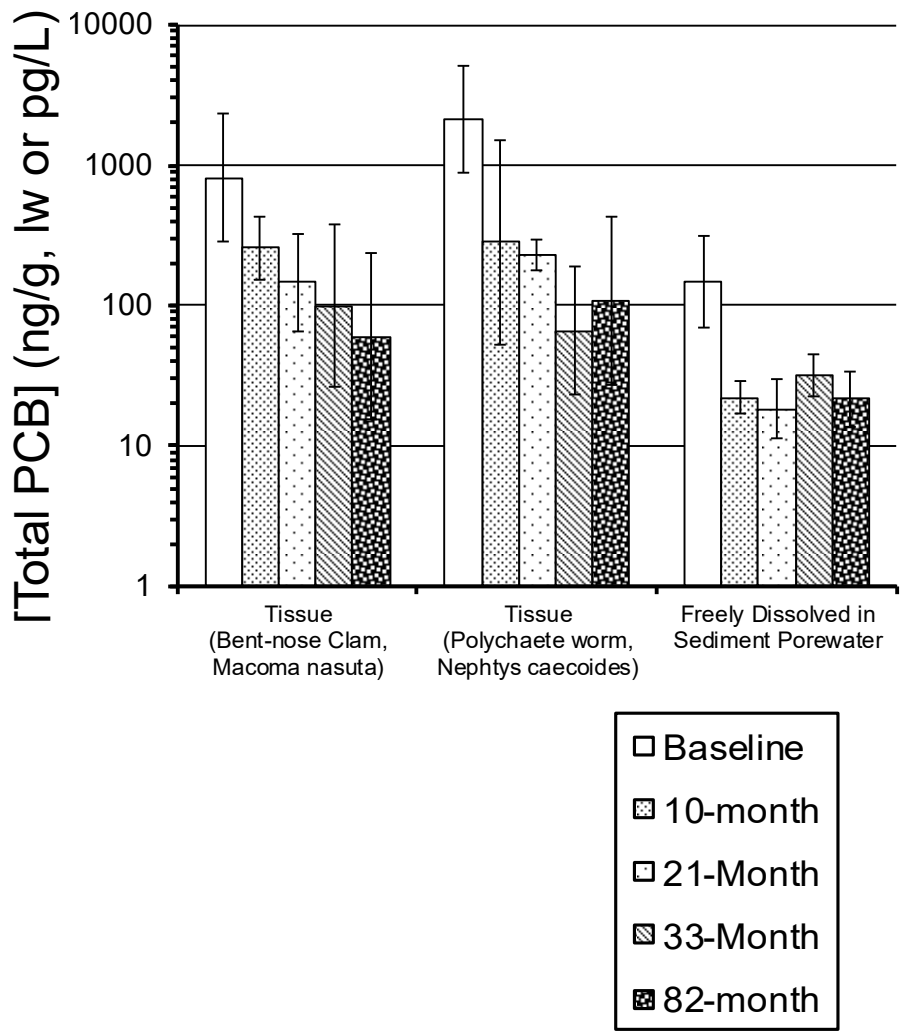
- No statistical differences among most metrics
  - Compare amended to baseline and reference
  - Abundance, Evenness, Dominance, Diversity
- One metric statistically lower at baseline unamended (reference) compared to 82-month amended
  - Suggests improvement in amended area
  - Taxa Richness
- Consistent with other studies suggesting no adverse effect on benthic community at AC dose rates <5%



Amendment: Present Absent

Key Points – AC Amendment

- Effective remedy for PCBs in active DoD harbor conditions ~7 years post placement
- Long-term (82-month post placement) performance verified
  - 80-90% reduction of PCBs in tissue and porewater
  - Continued absence of significant adverse impacts to benthic community
- Long-term stability, presence, and mixing verified
  - Using two methods for quantifying AC
  - Stability and mixing of the AC in the surface sediment at 82 months (~7 years) comparable to that at 33 months (~3 years)



Conclusions

- Design and placement timing relative to ship movements at deep water Navy sites like Pier 7
- Remedy performance
  - Long term effectiveness was originally estimated at >5 years
  - Amendment continues to perform at Pier 7 at ~7 years
- Remedy persistence
  - Aggregate and two methods of quantifying black and activated carbon suggest amendment present at 33-month levels
- Regulatory
  - Integrating reduced bioavailability metrics into remedial goals typically based on bulk sediment concentrations

Benefits to DoD

- Alternative to dredging of contaminated sediments
  - Less environmentally disruptive
  - Less costly
- Increased confidence in long-term performance of Activated Carbon-based remedies
- Data regarding stability, performance, and potential for adverse biological effects at an active, deep water Navy Shipyard at longer time points
  - Desired by relevant regulators and end users

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- Anne Christopher (EPA Region X)
- Leonard Machut (Washington Dept. of Ecology)
- NAVFAC Sediment Workgroup

[1]*Now with Exponent, Inc.*

For additional information, please visit

https://www.serdp-estcp.org/Program-Areas/Environmental-Restoration/ER18-5079

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Q&A Session 2

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