# Guidance on Passive Sampling Methods to Improve Management of Contaminated Sediments

Summary of a SETAC Technical Workshop

#### Edited by

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Summary of a SETAC Technical Workshop 7–9 November 2012 Southern California Coastal Water Research Project (SCCWRP) Costa Mesa, California, USA



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- US Department of Defense Strategic Environmental Research and Development Program (SERDP)
- Department of Environmental Sciences, University of California, Riverside

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### Introduction

#### Background and Need for the Workshop

Sediments serve as the sink for many contaminants that are released from domestic, agricultural, and industrial point and non-point sources. Contaminated sediments are found worldwide in many rivers, lakes, estuaries, and coastal areas and are often the consequence of historical practices of the early modern industrial era. Sediment-associated contaminants may directly impact benthic life as well as pose deleterious indirect effects on other organisms, including humans, via bioaccumulation and subsequent transfer through the food web. Regulatory agencies and responsible parties are increasingly tasked with identifying, investigating, and managing contaminated sediments. Management of contaminated sediments includes source and institutional controls, remediation, and evaluating effectiveness of the selected management actions. Past experience has shown that remediation is often difficult, costly, and time consuming, with predicted benefits realized only after long time frames. Contaminated sediments are recognized as a significant ongoing environmental problem that impacts the uses of many water bodies and as a contributing factor in many fish and shellfish consumption advisories.

To assess the potential impacts of contaminated sediments, contaminant analyses for bulk or whole sediments often serve as one critical line of evidence (LOE) used to support decision-making. These analyses typically involve exhaustive extraction methods that are intended to quantify "total" contaminant concentrations in the sediment. However, such measurements provide a poor predictor of exposure and subsequent risk because contaminant bioavailability is ignored. To address the bioavailability issue, equilibrium partitioning (EqP) models were developed to predict freely dissolved concentrations in sediment porewater, or  $C_{\text{free}}$ , for both organics and selected metals. The prediction of  $C_{\text{free}}$  is a proxy for the chemical activity of sediment contaminants that drives environmental processes. Implementation of these approaches is straightforward because bioavailability is assessed by normalizing total concentrations to the dominant binding phase (total organic carbon for non-ionic organics, acid volatile sulfide for divalent metals). While these approaches often work well in laboratory-spiked sediments and have thus offered an initial step forward, such normalization procedures often do not reliably characterize the bioavailability of contaminants in field sediments due to the presence of highly sorptive binding phases (e.g., black carbon) and sequestration processes. Given the cost implications of remedial decisions, these findings have led to a growing body of literature on the use of passive sampling methods (PSMs) that aim to quantify the bioavailability of both organics and metals in sediments. For the purpose of this workshop, passive sampling PSMs were broadly defined as techniques that quantify bioavailability based on the diffusion and subsequent partitioning of contaminants from sediment to a reference sampling phase.

#### 6 Passive Sampling Methods for Management of Contaminated Sediments

Despite significant advances in the development and application of PSMs over the past decade, incorporation of these tools in contaminated sediment management decisions has been limited. The primary barriers to the broader regulatory acceptance and use of PSMs are 1) failure of practitioners and decision makers to understand the advantages and limitations of these chemical-based approaches over traditional analytical methods and 2) confusion regarding the plethora of different methods and formats that are increasingly reported in the literature (e.g., PSMs can be deployed in the laboratory or field and applied under equilibrium or kinetic modes). Further barriers are lack of consensus on technical guidance for PSM selection, standardization, and use in regulatory decision-making contexts and limited experience by commercial laboratories in use and analysis of PSMs.

#### Purpose, Scope, and Goals

The purpose of this workshop was to promote understanding of PSMs and provide recommendations for current and future use in contaminated sediment management decisions. While PSMs can be applied to other media such as soil, groundwater, or air, the steering committee decided to focus on contaminated sediments given their present management challenges. The workshop scope covered freshwater, estuarine, and marine sediments as well as major contaminant classes (hydrophobic organic chemicals, metals including metalloids, and other inorganic contaminants of concern, including As and Se). The workshop comprised four separate workgroups:

The objective of Workgroup #1 was to summarize the literature describing types and uses of PSMs in contaminated sediments. The intent of this review was to cover both organics and metals and to serve as a resource upon which the other workgroups could draw.

The charge of Workgroup #2 was to describe the scientific rationale supporting PSM measurements as an improved basis for exposure and risk characterization of contaminated sediments. Thus, this workgroup was tasked with articulating the technical basis for advancing use of PSMs as a bioavailability-based LOE in decision-making.

The aim of Workgroup #3 was to provide practical guidance for laboratory and field deployment of PSMs. This guidance included the key considerations for PSM method selection, standardization, and quality assurance identified as key requirements for broader acceptance.

The objective of Workgroup #4 was to define current management applications and future opportunities, including research and communication needs for PSMs in contaminated sediment decision contexts.

#### Participation and Format

In late 2011, the workshop steering committee identified and contacted 45 international experts from business, academia, and government (see Appendix 1). Participants were selected to represent different affiliations, regions, genders, and stakeholder perspectives, including researchers (those who develop the PSMs and models), practitioners (industry, consultants, and regulators who will apply or manufacture PSMs in laboratory or field studies), and decision-makers and resource managers (end users of PSM data). Prior to the workshop, the general view among the steering committee was that, in the case of non-ionic hydrophobic organic chemicals (HOCs), PSMs have advanced to the stage that application in current decision-making is possible. However, in contrast to non-ionic organics, the opinion was that management application of PSMs for metals was largely a future opportunity. Consequently, while there was consensus that the focus of the workshop would be directed at HOCs, the steering committee agreed to include metals in the workshop scope. This decision is reflected in the fewer number of workshop participants with unique metals expertise.

Participants were assigned to one of the four workgroups described above. Beginning in April 2012, the Workshop Steering Committee held calls with workgroup members to develop draft manuscripts that would serve as initial deliverables for input to the workshop. Draft papers from each workgroup were to serve as the basis for discussion, debate, and consensus building at the workshop. In addition, a workshop agenda was developed to balance the need for inter- and intra-workgroup interactions (see Appendix 2).

The workshop was held on 7–9 November 2012 in Costa Mesa, California, USA. During the first day, each workgroup was afforded the opportunity in a plenary session to present key findings from their draft manuscript. Efforts by Workgroup #1 produced two manuscripts that highlighted PSM applications for organics and metals, respectively. The key findings of each workgroup were reviewed and discussed by all workshop participants. Collective feedback provided during the initial plenary session was then used to guide revisions to each draft manuscript during subsequent workgroup breakouts and follow-up plenary discussions that comprised the remaining workshop format.

One key point of discussion was the two general methodological approaches that have been used for quantifying the bioavailability of contaminants in sediments. The first approach relies on the concept of chemical activity, which aims at determination of  $C_{\text{free}}$  in interstitial water. A variety of PSM phases that have been used for this purpose were highlighted, including polydimethylsiloxane (PDMS), polysiloxane (silicone rubber) polyoxymethylene (POM), polyacrylate (PA), ethyl vinyl acetate (EVA), and low density polyethylene (LPDE) in different configurations (e.g., sheets, tubes, coated fibers, or vials). The second approach is based on the concentration that can be rapidly desorbed from the sediment using a commercial sorbent that serves as an infinite sink (e.g., Tenax beads or XAD

resin). Such desorption-based methods are empirically defined by the analytical protocol used to determine the bioavailable concentration. Participants agreed to limit focus at the workshop to chemical-activity based PSMs that target reliable measurement of  $C_{\rm free}$  for HOCs.

## **Preliminary Findings**

#### Workgroup 1 – State of the Science

Workgroup 1 addressed the following questions:

- Which passive sampling methods have been successful in representing the bioavailability of sediment-associated organic and trace metal contaminants?
- Which analytes have been targeted?
- What assessment endpoints have been evaluated?
- Have these methods been applied in lab and field situations?

*Hydrophobic organic compounds (HOCs)*. A comprehensive survey of peer-reviewed and "grey" literature revealed that a significant body of information details the calibration and application of PSMs for assessment of sediments contaminated with HOCs, including polycyclic aromatic hydrocarbons (PAHs) and chlorinated hydrocarbons such as polychlorinated biphenyls (PCBs and organochlorine biocides such as DDTs and chlordanes]). Polymers in multiple configurations that have been pre-calibrated for the aforementioned analytes of interest have been used to estimate  $C_{\rm free}$  and ultimately to predict endpoints such as bioaccumulation (i.e., tissue concentration), acute and chronic toxicity (LC50, EC50), and mass flux escaping from a contaminated sediment bed (Table 1; Figure 1), which inform management decisions.

However, the literature also demonstrates the wide range of calibration parameters that have been published for the various polymers and configurations of passive samplers. For example, values of log of the PDMS–water partition coefficient reported for the same PAH, PCB, and DDT analytes span a range of 1 to 3 orders of magnitude. Thus, expert consensus on standardization of calibration procedures and selection of appropriate calibration parameters for polymer phases and configurations is critical for ensuring accurate estimation of  $C_{\rm free}$ , a precursor to the successful application of PSMs.

More than 100 peer-reviewed papers that describe PSMs targeting the freely dissolved concentration of HOCs and  $C_{\text{free}}$  were compiled. These methods incorporate polymers as sorbing phases for HOCs or as ion exchange media (e.g., hydrogels) for trace metals (Table 1).

		•
Passive Sampling Phase or Media	Configuration	Target Analytes
Polydimethylsiloxane (PDMS)	Coated fiber, vial	HOCs
Polyethylene (PE)	Film/sheet, tube	HOCs
Polyoxymethylene (POM)	Film/sheet	HOCs
Ethylvinylacetate (EVA)	Coated vial	HOCs
Silicone rubber (SR)	Sheet, ring	HOCs
Gels	Diffusive gradient thin film (DGT)	Metals
Resin impregnated polyacrylamide gel	"Gellyfish"	Metals
Metal-chelating media	Disk/membrane	Metals
Water-filled equilibration cell	"Peeper"	Metals

Table 1: Passive sampling phases and configurations for organic and metal analytes

Metals. Trace metals such as Cd, Cu, Pb, Hg, Ni, and Zn constitute a special case with regard to the development and application of PSMs. Compared to HOCs, the fate, uptake, and toxicity of metals are subject to inherent fate- and effect-regulating processes that effectively change the character of metal speciation and interaction with biotic ligands. As a result, physiologically based trafficking pathways within organisms induce highly variable accumulation patterns from sediments of varying composition but with similar metal loading. Consequently, the scientific literature on the use of PSMs for sediment-associated metals is less established than for organics; to date such use is restricted to investigating metal digenesis and establishing relationships between PSMs and free-living or caged aquatic organism responses. As with organics, however, it has become generally accepted that freely dissolved concentrations of metals in sediment interstitial water  $(C_{\text{free}})$  provide a more relevant exposure metric for risk assessment than do total metal concentrations in bulk sediment. Moreover, PSMs offer promise for cost-efficient and accurate in-situ characterization of C<sub>free</sub>, allowing for detection of time-averaged exposures, and the ability to characterize episodic events and cyclic changes that may be missed by snapshot-in-time or grab sampling.

Like HOCs, successful application of PSMs for metals in a regulatory context will require standardization and tailored practical guidance, as well as the generation of compelling information showing benefits compared with and in addition to conventional risk assessment parameters. Equally important with regard to future acceptance of PSMs for metals is the ability to reconcile measurement results to geochemical speciation models that are currently applied to natural waters. Because regulatory decisions are increasingly based on model calculations, it will be imperative to understand the uncertainties associated with the most successful PSMs, as well as the discrepancies between PSM measurements and speciation model calculations.

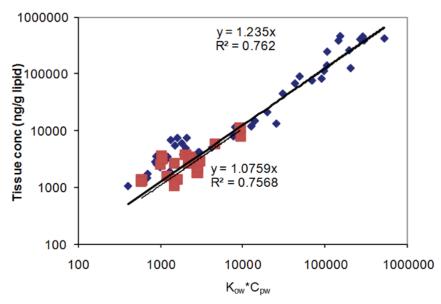


Figure 1. Comparison of predicted tissue concentrations in oligochaetes derived using passive samplers with measured data. Symbols denote river (squares) and diluted river (diamonds) sediments.  $K_{ow}$  = octanol-water partition coefficient;  $C_{pw} = C_{free}$ . (Source: Lu X, Skwarski A, Drake B, Reible DD. 2011. Predicting bioavailability of PAHs and PCBs with porewater concentrations measured by solid-phase microextraction fibers. Environ Toxicol Chem. 30(5):1109–1116. Reprinted with permission.)

#### Workgroup 2 - Scientific Rationale and Theoretical Considerations

Workgroup 2 addressed the following auestions:

- What is the scientific basis for incorporating PSMs to improve exposure and risk characterization of contaminated sediments?
- What are the key principles that will lead to successful application of PSMs in contaminated sediment assessment and management?

Members of Workgroup 2, invoking the basic principles of chemical thermodynamics, recognized that chemical activity (A) is superior to bulk or "total" concentration in describing the bioavailability of HOCs and metals in sediments. While the workgroup reached consensus on the utility of chemical activity as a unifying concept, the members also recognized the advantages of translating activity-based measurements into  $C_{\rm free}$  to facilitate communication and acceptance of PSM data. At thermodynamic equilibrium, the chemical activity across environmental compartments (e.g., sediment, aqueous phase, biological lipid, and a passive sampler polymer phase) is by definition equal, and  $C_{\rm free}$  is directly related to the concentration in the passive sampler polymer phase ( $C_p$ ) (Figure 2). Thus while the mass of contaminants in different phases may vary depending on the phase capacity, the activity across phases is constant. This approach involves measuring the equilibrated polymer concentration and subsequent translation to  $C_{\rm free}$  using the substance-specific polymer–water partition coefficient ( $K_{\rm pw}$ ). Thus,  $C_{\rm free}$  is not measured directly and thus depends on accurate  $K_{\rm pw}$  values for the substance of concern.

For PSMs to be successful, however, two critical conditions must be met: 1) attainment of equilibrium (or near-equilibrium) should be achieved and 2) PSMs should not result in local depletion or concentration of the target HOC, disrupting the pool of contaminant available for exchange across the compartments present (Figure 2). In the absence of equilibrium (or near-equilibrium), correction to an equilibrium condition using performance reference compounds (PRCs) can be performed, assuming reliable, validated methods for such correction are available.

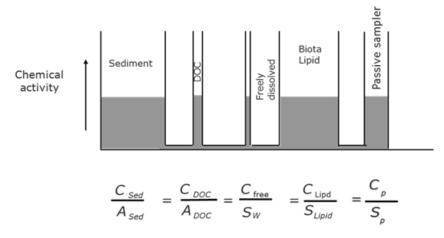


Figure 2. Conceptual diagram illustrating that the passive sampler detects the activity of the sampled environment. *S* denotes the solubility of the chemical in the respoective phase.

Key relationships between  $C_{\rm free}$  and endpoints of interest, including bioaccumulation by organisms of interest, toxicity as represented by the lethal or chronic effect concentration, and HOC flux from sediments with a gradient of contamination, were also established. For bioaccumulation, the workgroup recognized that PSMs can address potential bioconcentration by benthic organisms and also, with the appropriate model construct, indirect transfer via the food web.

# Workgroup 3 – Practical Guidance for Application in Laboratory and Field Settings

Workgroup 3 addressed the following questions:.

- How should PSMs be implemented?
- How will methods be calibrated or validated?
- What is the uncertainty associated with the parameters defined using PSMs?

Workgroup 3 agreed that several PSMs identified by Workgroup 1 were ready for application; however, the group could not reach consensus on guidance for practitioners on which methods should be used and how these methods should be implemented and validated, and additional development is required. In response, the following set of guiding principles were developed for selection, preparation, implementation, and validation of PSMs, starting with translation of the management question into measurement goals for  $C_{\rm free}$  and ending with a discussion of the uncertainty:

1) Practical guidance (e.g., selection of PSM) is driven by the question asked.

#### Step 1: Define the management need and generate measurement goals for $C_{\text{free}}$ .

- 2) Key considerations should be addressed when a PSM-based solution is selected and designed. These considerations can be grouped broadly as:
  - a) technical (analytes of interest, sensitivity, accuracy, and precision of measurements, spatial or temporal resolution)
  - b) non-technical (site accessibility, cost, level of expertise required, availability of method via the commercial services community)

Step 2: Perform trade-off of key considerations to select the most appropriate PSMs.

3) Both ex-situ and in-situ applications can be used to address management questions.

# Step 3: Determine the pros and cons of ex-situ or in-situ application for the problem context.

- 4) QA/QC is critical for validation of  $C_{\text{free}}$  and, at a minimum, should include:
  - a) selection and use of appropriate pre-calibration parameters (e.g.,  $K_{\rm pw}$  values)
  - b) provisions to ensure attainment of equilibrium, or alternatively, for correction to an equilibrium condition
  - c) guidelines to ensure non-depletive conditions

#### Step 4: Establish QA/QC guidelines commensurate with project goals.

5) Sources of uncertainty in measuring and applying  $C_{\text{free}}$  in subsequent risk assessment exercises are understood and will vary based on the application scenario (e.g., in situ versus ex situ). Our current understanding of uncertainty associated with  $C_{\text{free}}$  measurement using PSMs is expected to be only a fraction of the uncertainty associated with the status quo (i.e., organic normalization bulk sediment concentration as a primary exposure metric to infer  $C_{\text{free}}$ ).

Step 5: Quantify PSM measurement uncertainty and propagate through the assessment process.

#### Workgroup 4 – Management Applications

Workgroup 4 addressed the following questions:

- What are the current and projected future management applications for PSMs in assessing and managing risk associated with contaminated sediments?
- What is needed for widespread acceptance of PSMs among the management community?
- What research and technology developments will support broader application of PSMs?

Passive sampling methods that target  $C_{\text{free}}$  give managers a better predictor of bioavailability than the status quo (i.e., bulk sediment or total concentration) for 3 key exposure pathways: direct exposure to invertebrates with respect to either toxicity or bioaccumulation, flux from sediments to the overlying water column, and exposures within the water column (Figure 3). Ex-situ or in-situ application of PSMs to measure  $C_{\text{free}}$  relative to these 3 pathways will reduce uncertainty in sediment risk assessment and subsequent risk management decisions over the full range of spatial and temporal scales. The  $C_{\text{free}}$  LOE can be used alone for screening or along with other LOEs in a weight of evidence (WOE) assessment.

The  $C_{\text{free}}$  LOE can be used in determining contaminant sources, guiding remedial decisions (defining remedial zones, evaluating remedial options, and remedial design), and monitoring short- and long-term success of specific remedies (i.e., assessing changes in bioavailable contaminants). PSM-derived  $C_{\text{free}}$  measurements can also be used for modeling future conditions. Additional utility is expected as PSM use is optimized and improved, and as new technologies such as omics and remote sensing networks come on-line.

To gain widespread acceptance of PSMs among managers, scientists and practitioners must work together to seamlessly incorporate  $C_{\text{free}}$  into existing risk

assessment frameworks, and to create education and outreach opportunities on the appropriate and inappropriate uses of this LOE.

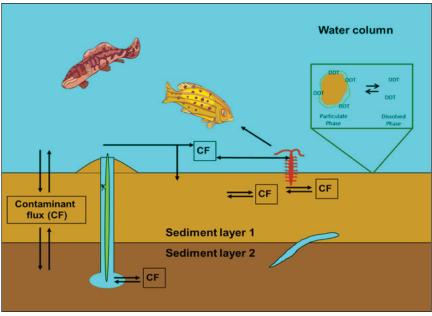


Figure 3. Conceptual site model that can be linked to Cfree measurements

### **Overall Workshop Summary**

Assessing and managing risks associated with contaminated sediments remains a challenge. Current practice relies on LOEs, including total chemical concentrations in bulk sediments, and equilibrium partitioning models that inaccurately address differential bioavailability among field sediments. PSMs have evolved as an effective approach for measuring  $C_{\rm free}$  of target chemicals in sediments that can be related to endpoints of interest to management. The workshop participants agreed that PSMs provide a developing but better alternative to the status quo and that several methods are ready for application to support improved management of contaminated sediments. However, practical application of PSMs ultimately is driven by the question to be addressed. Selection and implementations. Several existing management applications for PSMs were identified as well as a number of future recommendations that would expand and enhance the use of these methods.

## **Next Steps**

The proceedings of the workshop will be submitted as a Special Series of papers to the SETAC journals *Environmental Toxicology and Chemistry (ET&C)* and *Integrated Environmental Assessment and Management (IEAM)*.

The proposed titles of these papers are:

- Passive Sampling in Contaminated Sediment Assessment: Overview of Workshop Goals and Deliverables to *IEAM*
- Passive Sampling in Contaminated Sediment Assessment: Literature Review for Non-ionic Organic Substances – to *ET* C
- Passive Sampling in Contaminated Sediment Assessment: Literature Review for Metals to  $ET \not \sim C$
- Passive Sampling in Contaminated Sediment Assessment: Practical Considerations for Lab and Field Deployment to *IEAM*
- Passive Sampling in Contaminated Sediment Assessment: Management Applications to *IEAM*

It is the intent of workshop participants that these papers will promote a greater understanding and use of PSMs in management of contaminated sediments.

# Appendix 1 – Workshop Participants

Name	Affiliation	Country	Work- group
Rachel Adams	Loyola Marymount University	USA	2
Ian Allan	Norwegian Institute for Water Research	Norway	4
Mayumi Allinson	University of Melbourne	Australia	1
Kim Anderson	Oregon State University	US	4
Sabine Apitz	SEA Environmental Decisions Ltd	UK	4
Chris Beegan	California Water Resources Control Board	USA	4
Todd Bridges	U.S. Army Corps of Engineers	USA	4
Steven Brown	The Dow Chemical Company	USA	2
Robert Burgess	U.S. Environmental Protection Agency	USA	3
Peter Campbell	Universitie du Quebec, INRS	Canada	1
John Cargill	Delaware Department of Natural Resources	USA	2
Peter Chapman*	Golder Associates Ltd	Canada	4
Yongju Choi	Stanford University	USA	3
Beate Escher*	University of Queensland	Australia	2
Will Gala	Chevron	USA	3
Jay Gan	University of California Riverside	USA	2
Upal Ghosh*	University of Maryland Baltimore County	USA	3
Frank Gobas	Simon Fraser University	Canada	3
Todd Gouin	Unilever	UK	2
Marc Greenberg*	U.S. Environmental Protection Agency	USA	4

Name	Affiliation	Country	Work- group
Phillip Gschwend	Massachusetts Institute of Technology	USA	2
Amanda Harwood <sup>#</sup>	Southern Illinois University	USA	1
Steven Hawthorne	Energy and Environmental Research Center	USA	2
Paul Helm	Ontario Ministry of the Environment	Canada	2
Michiel Jonker	Institute for Risk Assessment Sciences	Netherlands	3
Susan Kane-Driscoll*	Exponent	USA	3
Peter Landrum*	National Oceanic and Atmospheric Administration (ret.)	USA	1
Huizhen Li <sup>#</sup>	Guangzhou Institute of Geochemistry	China	1
Michael Lydy*	Southern Illinois University	USA	1
Keith Maruya*^	Southern California Coastal Water Research Project	USA	1,3
Philipp Mayer*	Aarhus University	Denmark	2
Megan McCulloch	Sediment Management Work Group	USA	4
Charles Menzie	Exponent	USA	4
Julie Mondon	Deakin University	Australia	1
Munro Mortimer	University of Queensland	Australia	3
Jochen Mueller	University of Queensland	Australia	NA
Amy Oen	Norwegian Geotechnical Institute (NGI)	Norway	1
Thomas Parkerton*^	ExxonMobil Biomedical Sciences Inc	USA	2,4
Willie Peijnenburg*	National Institute Public Health and Environment (RIVM)	Netherlands	1

Name	Affiliation	Country	Work- group
Danny Reible	University of Texas	USA	3
James Shine	Harvard University	USA	4
Foppe Smedes	Deltares	Netherlands	1
Gesine Witt	University of Applied Sciences Hamburg	Germany	2
Jing You	Guangzhou Institute of Geochemistry	China	2
Eddy Zeng*	Guangzhou Institute of Geochemistry	China	NA

^ co-organizer; \* Steering Committee; # student participant; NA - not assigned

## Appendix 2 – Workshop Agenda

Guidance on Passive Sampling Methods to Improve Management of Contaminated Sediments

Costa Mesa, California, USA, 7–9 November 2012

#### Wednesday, 7 November

Opening plenary (all participants)

Welcome and introduction – K. Maruya, T. Parkerton Workgroup 1 – State of the Science – M. Lydy, W. Peijnenburg Workgroup 2 – How to Best Utilize PSMs – P. Mayer, B. Escher Workgroup 3 – Practical Guidance – U. Ghosh, S. Kane-Driscoll Workgroup 4 – Management Applications – P. Chapman, M. Greenberg Guidance for breakout sessions – K. Maruya, T. Parkerton

Workgroup breakout session (concurrent)

#### Thursday, 8 November

Workgroup breakout session (concurrent)

Day 2 plenary (all participants)

Review workgroup progress (workgroup chairs)

#### Friday, 9 November

Workgroup breakout session (concurrent)

Closing plenary (all participants)

Workshop discussion on consensus and non-consensus items Summary, actions, schedule, and wrap-up

# SETAC

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- Science-based objectivity

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