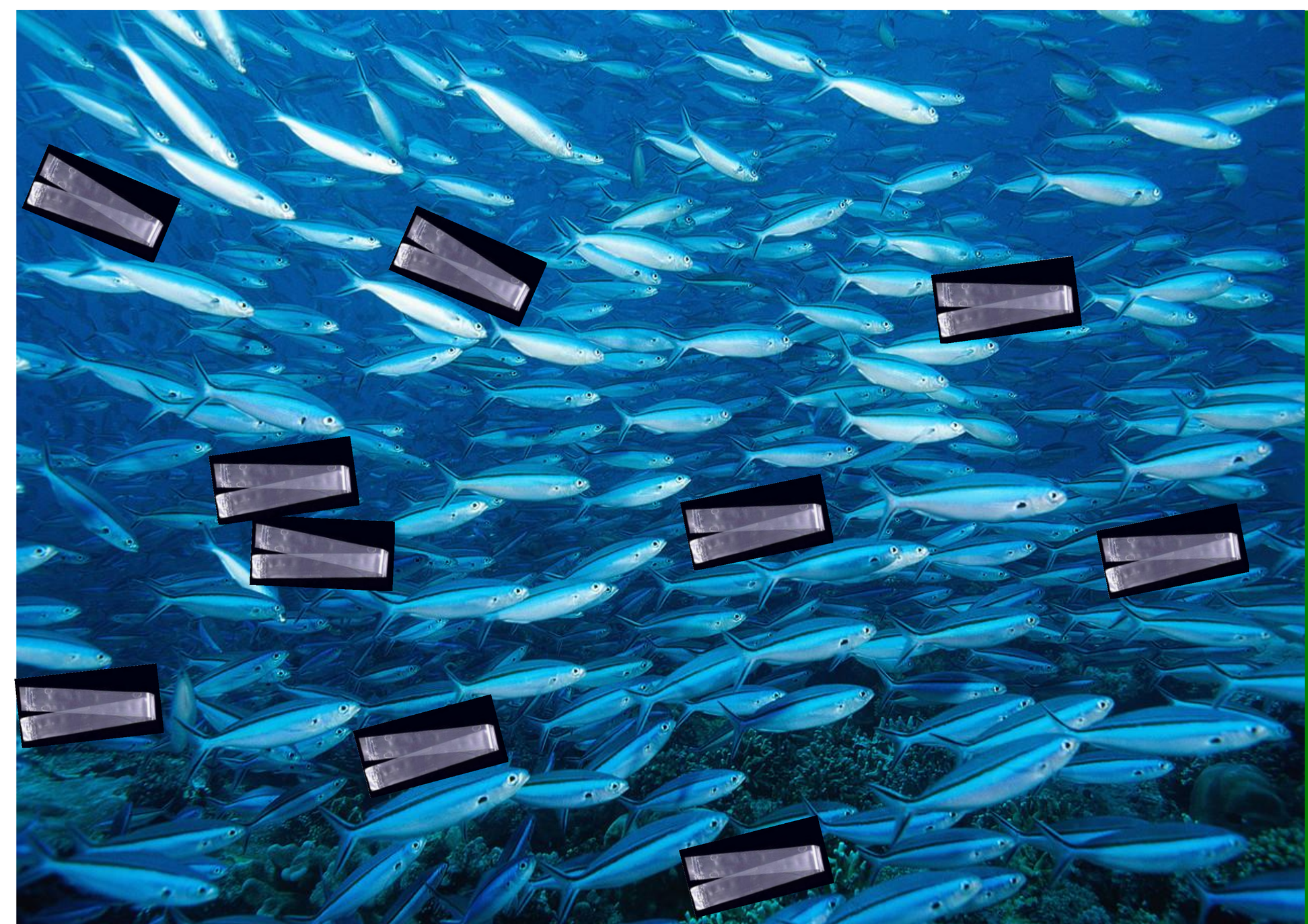

SOPs for SPMDs

http://ecy.wa.gov/programs/eap/quality.html

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Planning | Field Work | Analysis | Data Reduction | Quality Control | Data Management

Washington State Department of Ecology (Ecology) developed Standard Operating Procedures (SOPs) describing tasks for studies using semi-permeable membrane devices (SPMDs) to monitor hydrophobic organic compounds in surface water.

The *SOP for Conducting Studies Using SPMDs* is for managing project information and data collected prior to lab analyses.

The *SOP for SPMD Data Management and Data Reduction* directs procedures for after the field work.

Both SOPs follow guidelines developed by USGS (Huckins et al., 2006 and Alvarez, 2010).

Project Planning

Project planning is an iterative process beginning with general goals and ideas and then refining these to define specific objectives and needs at appropriate levels of detail. Careful planning and attention to detail are needed in all phases of using SPMDs in order for data to meet project objectives.

Figure 1. Sample Site Reconnaissance.

Life of a SPMD	
Pre-Field Fabrication and Preparation	Manufacture PE Tube and Lipid
	Spiking PRCs
	Storage at EST, Transport to Ecology – Field
Field Use	Deployment into Water
	In Water: Targeted Sampling Event
	Retrieval from Water
	Transport to and Storage at EST
Post-Field Processing and Extraction	Spiking: Surrogates (pesticides, PAHs, PBDEs) and PCB EIS
	Extraction (dialysis)
	Cleanup: GPC
	Extract Splitting
	Storage and Transport to Analysis Labs
Lab	Extract Splitting
	Extract Analysis

Table 1. Overview of the Life of a SPMD.

Site Considerations include: the ever popular ubiquitous NorthAmericanesFishermanes is attracted to shiny things and leads to loss of and or damage to deployment canisters. (Cranor, W. 2008. USGS Chemist. Personal Communication).

Master Analysis and Sampling Plan

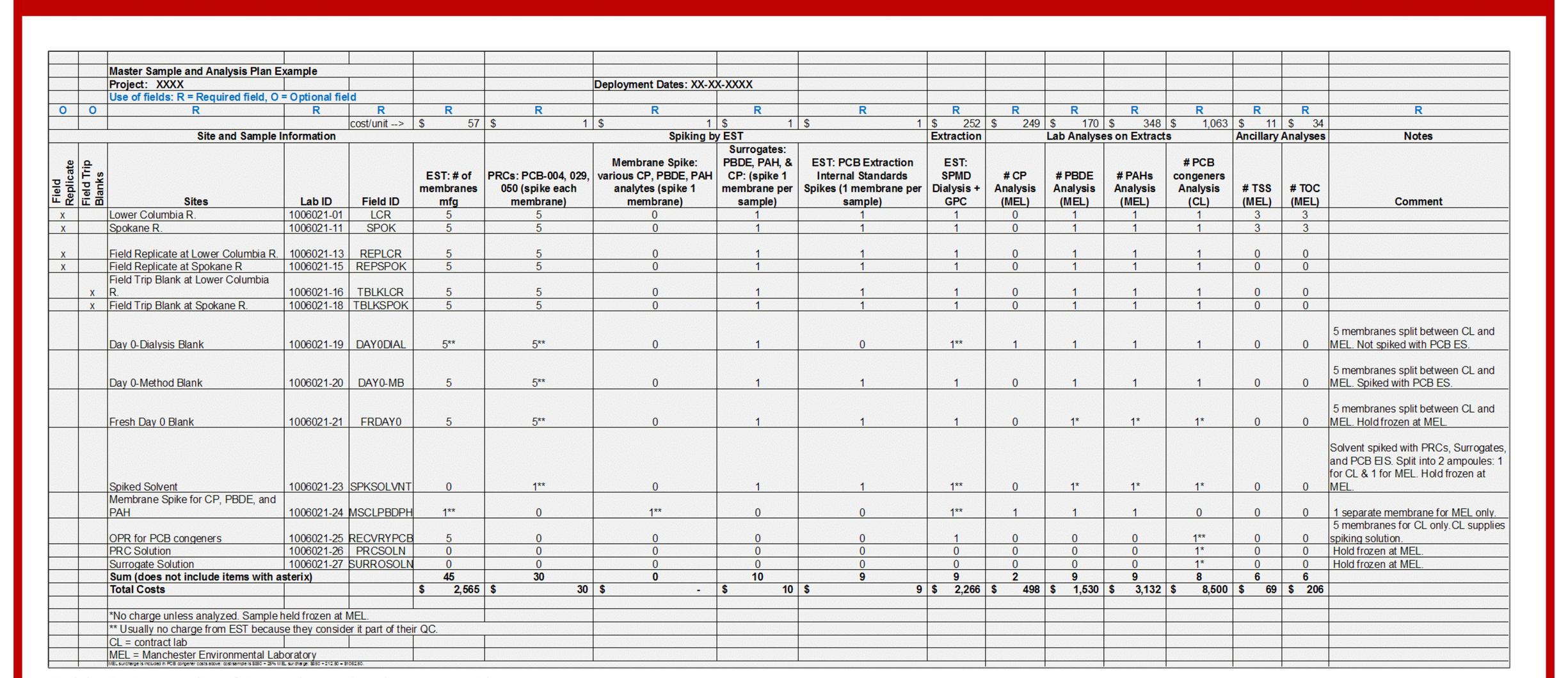


Table 2. Example of Sample and Laboratory Plan.

Clearly identify and communicate actions at each step.

Deployment

Pre-Field Deployment

- Consider site variations and review deployment techniques.
- Prepare equipment through inventory, purchasing, cleaning, and calibrating.
- Prepare field folders and obtain permission for site access.

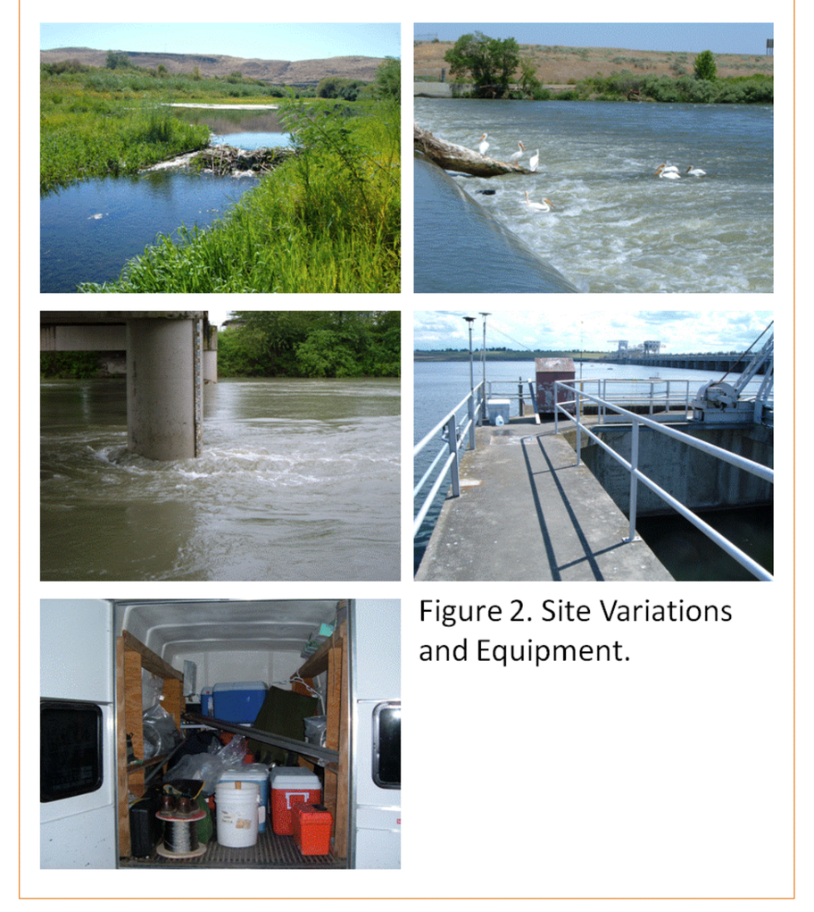


Figure 2. Site Variations and Equipment.

Field Deployment

- Prepare deployment assembly.
- Deploy SPMDs.
- Check samplers midway through sampling period as an option.

Figure 3. Deployment Variations.

To address the possibility of the SPMDs getting compromised, a TidbiT™ temperature monitor was attached to the canister (a) and another to a structure (b) nearby for monitoring water and air temperature.

Figure 4. TidbiTs™ Attached to Canister and Bush.

Retrieval

Retrieve SPMDs From the Field

- Make sure the SPMD cans are labeled properly: date, sample ID, and number of membranes in each can.
- Remember, safety first.

Be prepared because conditions can change during deployment and make retrieval…

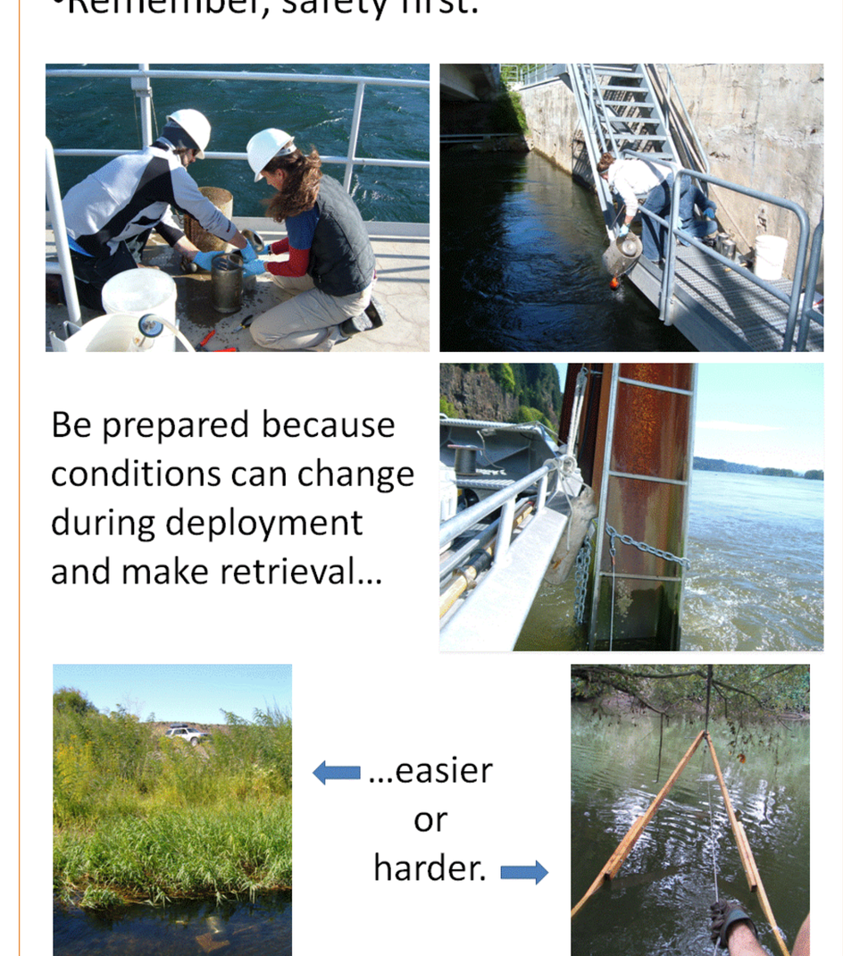
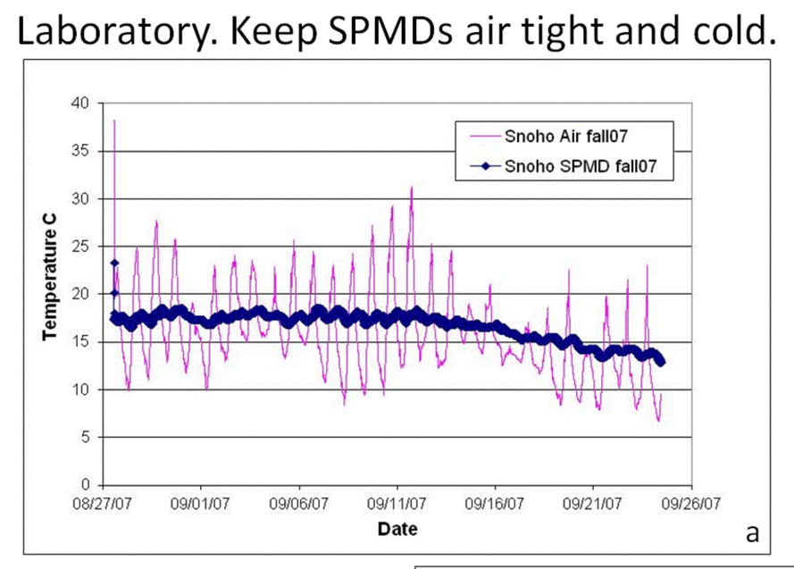


Figure 5. SPMD Retrievals.

Post-Field Deployment

- Review air and water temperature data (TidbiT™) to ensure integrity.
- Ship SPMDs to Environmental Sampling Technologies (EST) Laboratory. Keep SPMDs air tight and cold.



The air temperature (pink) shows the daily diurnal temperature variation while the water temperature (blue) shows a more consistent temperature.

SPMDs Not Compromised.

SPMDs Compromised.

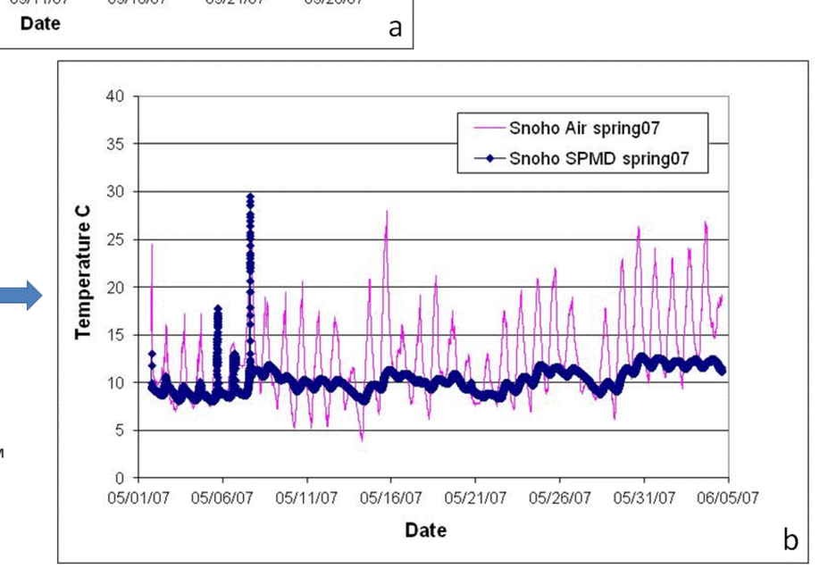
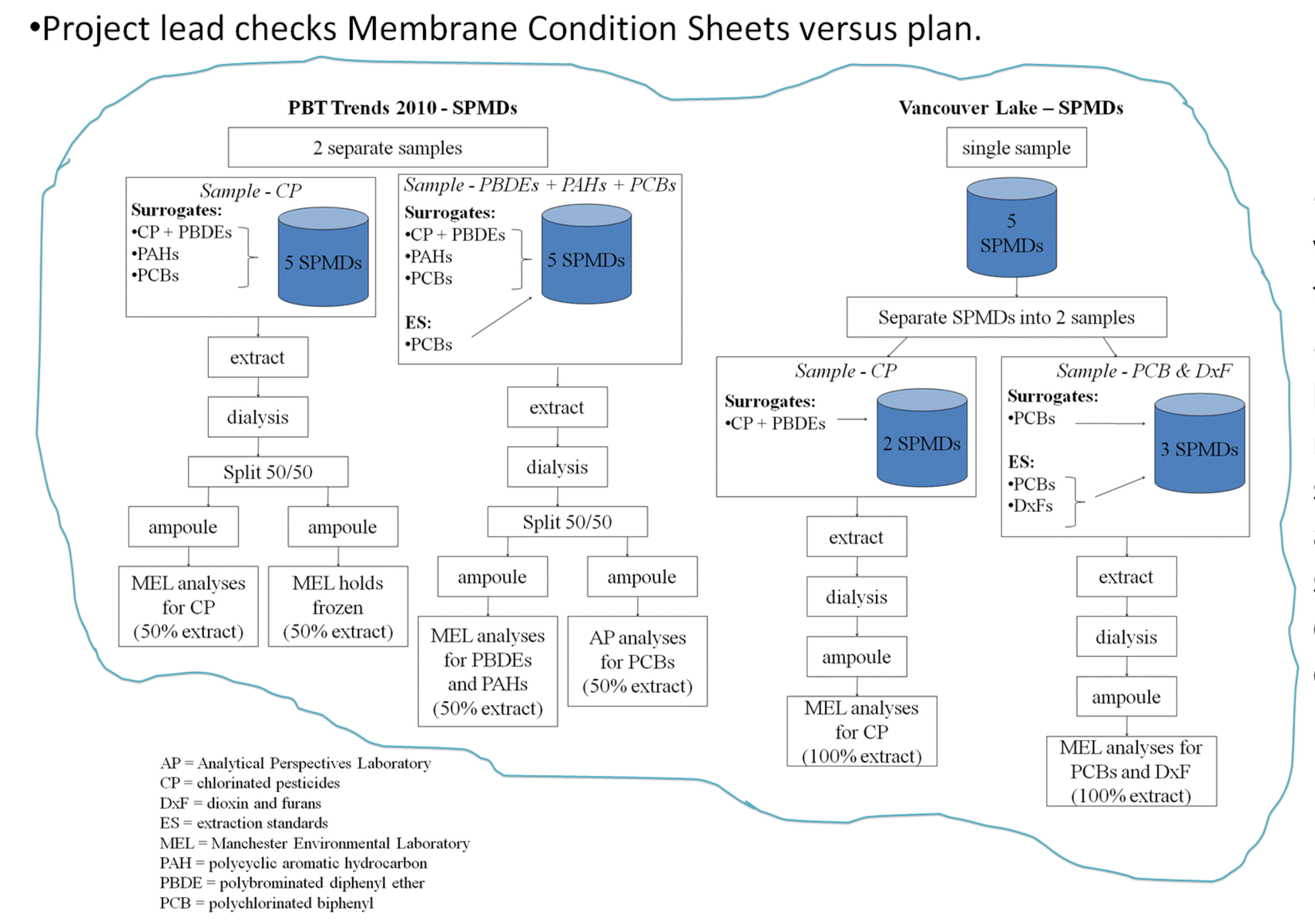


Figure 6 a and b. TidbiT™ Air and Water Data Compared.

Sample Processing and Analyses

Extraction and Dialysis

- EST performs post-field processing, spiking, extraction, splitting, ampulizing.
- **Critical**: Project lead cross-checks for correct spiking from all parties according to master plan; check math.
- Project lead checks Membrane Condition Sheets versus plan.



PBT Trends 2010 - SPMDs
2 separate samples
Sample - CP
Surrogates: •CP + PBDEs •PAHs •PCBs
5 SPMDs
Sample - PBDEs + PAHs + PCBs
Surrogates: •CP + PBDEs •PAHs •PCBs
ES: •PCBs
5 SPMDs
extract
dialysis
Split 50/50
ampoule
MEL analyses for CP (50% extract)
MEL holds frozen (50% extract)
MEL analyses for PBDEs and PAHs (50% extract)
AP analyses for PCBs (50% extract)
Vancouver Lake – SPMDs
single sample
5 SPMDs
Separate SPMDs into 2 samples
Sample - CP
Surrogates: •CP + PBDEs
2 SPMDs
Sample - PCB & DxF
Surrogates: •PCBs
ES: •PCBs •DxFs
3 SPMDs
MEL analyses for CP (100% extract)
MEL analyses for PCBs and DxF (100% extract)
AP = Analytical Perspectives Laboratory
CP = chlorinated pesticides
DxF = dioxin and furans
ES = extraction standards
MEL = Manchester Environmental Laboratory
PAH = polycyclic aromatic hydrocarbon
PBDE = polybrominated diphenyl ether
PCB = polychlorinated biphenyl

Laboratory Analyses

- Physical SPMD processing yields extract that goes to labs for analysis.
- The project lead checks laboratory reports (case narratives) for proper sample splitting, appropriate cleanup and analyses, outliers, spurious values, completeness of work and records, and others.

Figure 7. Example of Two Post-Field Pathways for SPMDs From Different Studies for Laboratory Analyses—each using multiple laboratories.

The Data Reduction procedure is listed in the *Data Management and Data Reduction* SOP. A detailed table was developed to be used as a guide, checklist, and place to document data reduction actions: it is not an instructional guide for the use of SPMDs.

Summary of Data Reduction Procedures

The "Data Reduction Tasks for SPMD Data" table is organized into Phases (A-M), Step #s, Tasks, and Notes. These guide SPMD users through all aspects of an SPMD study. Each Phase is a major task or action which is further divided into discreet "Steps", then tasks with associated actions, and finally notes and completion dates.

Phase	Steps	SPMD Processing Tasks	Project Officer Tasks in: QC, Checks, Data Reduction, Documentation	Project Officer Note	Project Officer Complete Date	Auditor Note	Auditor Complete Date	Relevant Appendix or Record

Table 2. Column Headings in "Data Reduction Tasks for SPMD Data" Table.

Major procedures for the Project Officer after laboratory analyses include:

- **Phase G:** reviews all Case Narratives.
- **Phase H:** reviews data sets.
- **Phase I:** evaluates blanks for contamination: Field, Process, and Reagent Blanks.
- **Phase J:** determines Limit of Detection (LOD) and Limit of Quantitation (LOQ) for each result using results from Field Blanks and decides whether to censor or blank correct.
- **Phase K:** estimates dissolved concentrations using USGS model.
- **Phase L:** determines whether and where to use summed or individual results for analyte groups (e.g. PCBs, DDTs, PBDEs, Chlordanes).
- **Phase M:** estimates total or whole water concentrations from model output and TOC data.

Figure 8. Evidence of a Beaver Performing Data Reduction With Water Data.

Guidance for determining detection and quantitation limits and background correction are addressed within Ecology's SOPs following the guidelines from USGS and referencing Keith (1991).

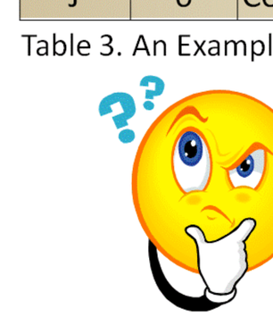
Ecology recommends a minimum of three field blanks for each SPMD study: preferably, one field blank at each site to characterize contamination in the sampling and measurement system.

Censor or Blank Correct

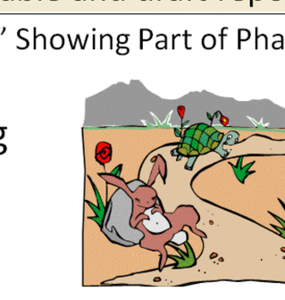
Phase J in the "Data Reduction Tasks for SPMD Data" gives two approaches for handling residue results: censoring or blank-correcting. Both approaches require the LOD and LOQ to be determined. The analytical laboratories cannot determine the LOD and LOQ because the extraction of the sample is performed at EST, not in the lab that analyzes the extract. Qualifiers assigned are for residues only and will help determine the qualifiers that are used for modeled water concentrations.

Phase	Steps	SPMD Processing Tasks	Project Officer Tasks in: QC, Checks, Data Reduction, Documentation
J	1	Determine LOD for each results and add to residue results table.	LOD = Mean of Field Blanks + 3 Std Devs. of the Mean. Set NDs = RL, or explain approach.
J	2	Determine LOD for each results and add to residue results table.	LOQ = Mean of Field Blanks = 10 Std Devs. of the Mean. Set NDs = RL, or explain approach.
J	3	Select one of two options for handling results: censor or blank correct.	Note that qualifiers are for residues only – and need to be retained for record-keeping.
J	4	1 - Censor: If the reported residue result "R" > LOQ, use the reported value. If R < LOQ, censor as UJ. The value associated with the UJ qualifier will be the LOD if R < LOD; or R if R is > LOD. Do not blank correct.	Document in residue data table.
J	5	2 - Blank Correct: If R > LOD, blank correct thus: R minus the mean of field blanks = blank corrected value. Qualify this value as "B1" which is defined as "Analyte detected in sample and method blank. Reported result is blank-corrected" (definition from EIM reference table). If R < LOD, qualify value as U.	ID and flag results that will be blank-corrected; and those that won't.
J	6	Consolidate and document in residue data table and draft report.	

Table 3. An Example of the "Data Reduction Tasks for SPMD Data" Showing Part of Phase J.



Allow enough time for working through the data reduction processes.



Abbreviations:
LOD: Limit of Detection.
LOQ: Limit of Quantitation.
Std Devs.: standard deviations.
ND: nondetected results.
RL: reporting limit.
R: residue result.
ID: identify.
EIM: Environmental Information Management (Ecology's electronic data storage).
UJ: analyte was not detected at or above the reported estimate.

Quality Control and Quality Assurance

Quality control and Quality Assurance practices for the *Conducting Studies Using SPMDs SOP* are described for three general locations where SPMDs and extracts are handled:

1. Field: ensuring sample integrity and use of replicate and field blank samples.
2. EST: characterizes contamination at various stages in the manufacturing and processing of SPMDs.
3. Analytical laboratories: variety of method-specific internal QC practices. Coordination among laboratories by project lead is imperative.

Quality Control and Quality Assurance for the *SPMD Data Management and Data Reduction* SOP consists of a review, or audit, of the SPMD project. The audit is an independent review to help ensure that the project was conducted using appropriate and defensible practices to support a credible set of data and report product.

The auditor checks work, records findings, and reports:

- Presence/absence and proper location of required records.
- Review of specific steps (at least 2-5) in more detail within each phase of the Data Reduction process.
- Completeness of records.

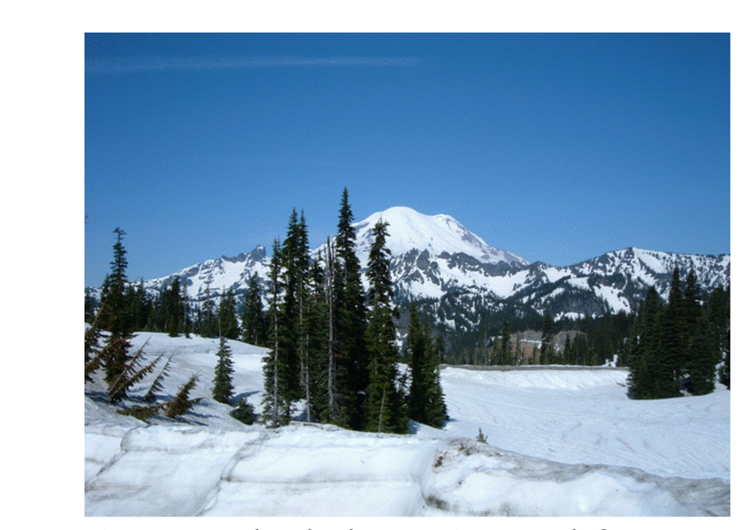


Figure 9. Rather be here or in an audit? Choices, Choices.

Figure 10. Shedding Some Light on the Subject.

SPMD Data Repository

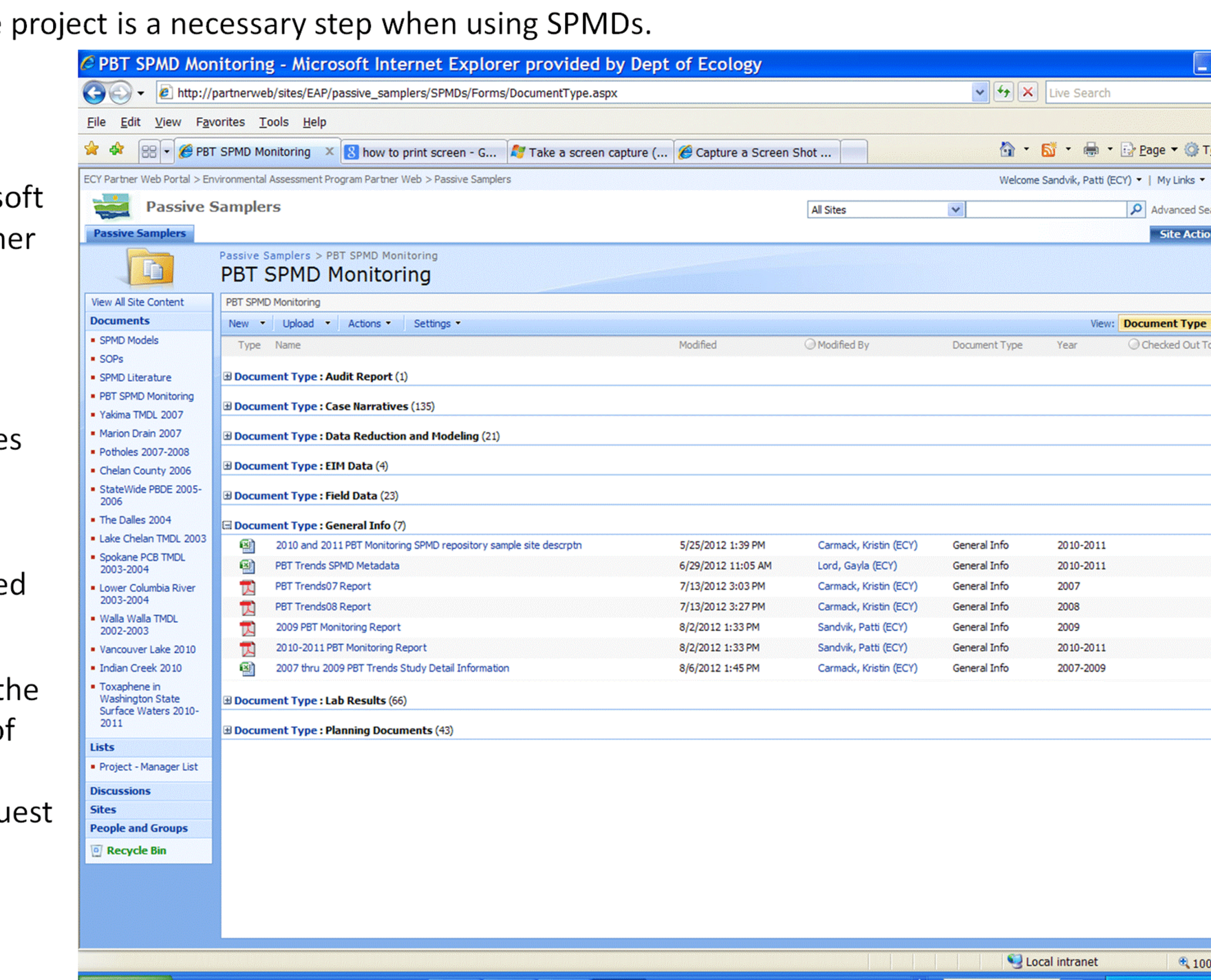
A data repository created for SPMD projects to house modeled data and all the supporting information generated during the project is a necessary step when using SPMDs.

Ecology has created a Passive Sampling repository in a Microsoft SharePoint Web Partner site.

The Passive Sampling SharePoint site houses individual projects in document libraries.

Editing access is limited for security .

Data are available to the public upon request of the project lead or Ecology's records request personnel.



PBT SPMD Monitoring - Microsoft Internet Explorer provided by Dept of Ecology
Passive Samplers
PBT SPMD Monitoring
Document Type : Audit Report (1)
Document Type : Case Narratives (135)
Document Type : Data Reduction and Modeling (21)
Document Type : EIM Data (4)
Document Type : Field Data (23)
Document Type : General Info (7)
Document Type : Lab Results (66)
Document Type : Planning Documents (43)

Table 5. Example of the Structure of Repository for SPMD Study Data.

Summary

Project leads need to manage large amounts of information in order to analyze, document, model, and report results. Careful thought and documentation must accompany the decisions and detailed actions for each step of the SPMD project.

References

Alvarez, D.A., 2010. Guidelines for the use of the semipermeable membrane device (SPMD and polar organic chemical integrative sampler (POCIS in environmental monitoring studies: U.S. Geological Survey, Techniques and Methods 1-D4, 28p.

Huckins, J.N., J.D. Petty, and K. Booij, 2006. Monitors of Organic Chemicals in the Environment; Semipermeable Membrane Devices: new York, Springer.

Keith, L.H., 1991. Environmental Sampling and Analysis; a Practical Guide: Michigan, Lewis Publishers, Inc.

Final SOPs for Conducting Studies Using Semipermeable Membrane Devices (SPMDs) and Data Management and Reduction can be found at www.ecy.wa.gov/programs/eap/quality.html.

DEPARTMENT OF ECOLOGY State of Washington

This poster is available on the Web at https://fortress.wa.gov/ecy/publications/SummaryPages/1203046.html. It was presented at the 5th International Passive Sampling Workshop and Symposium, September 11–12, 2012 in Columbia, MO.
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