Environment and Climate Change Canada
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Standard Operating Procedures for Management and Processing of Water Quality Data Collected using Semi-permeable Membrane Devices (SPMDs)

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Acronyms

COC	Chain of custody
ECCC	Environment and Climate Change Canada
EST	Environmental Sampling Technologies
ETI	Estimated time-integrated
FWQMS	Freshwater Quality Monitoring and Surveillance
PAC	Polycyclic aromatic compounds
PRC	Performance reference compound
QA	Quality assurance
QC	Quality control
RL	Reporting limit
SOP	Standard operating procedure
SPMD	Semi-permeable membrane device
USGS	United States Geological Survey

Definitions

Analyte – A compound occurring in a sample and quantified in a laboratory.

Background – Typical concentrations of analytes within sampling equipment (semi-permeable membrane devices [SPMDs], blanks) prior to targeted environmental exposure or concentrations to which samples are exposed within the laboratory environment, and which artificially raise analyte concentrations within the environmental sample.

Calibrated – Normalized to site-specific conditions such that concentrations may be compared between sites and over time. Concentrations of performance reference compounds (PRCs) prior to (initial) and after deployment (final) are used to calculate the rate of loss of organic compounds, which may be affected by water temperature, turbidity, biofouling, flow velocity, and turbulence.

Case Narrative – A written record of sample-specific analytical issues or mishaps and a description of the resulting bias in data measures.

Chain-of-Custody – Document that remains with a sample and is used to document details of transfers of the sample from the custody of one responsible body (custodian) to the custody of another.

Companion Sample – Water and field blank samples that are collected from the same site, at the same time, are referred to as “companion” samples.

Composite Sample – SPMD sample (see “Sample”) that consists of multiple membranes treated identically in the field and the lab. Dialysates are combined and homogenized prior to chemical analysis.

Conventional Water Quality Monitoring – Water quality monitoring program built upon

the collection of discrete (i.e., single point in time) samples rather than time-integrated passive samples.

Conversion / Converted Data – Use of the USGS Estimator to convert raw data (ng/SPMD) to estimated, time-integrated, dissolved concentrations (ng/L) that are calibrated to site-specific conditions. These normalized data are considered comparable between sites and over time.

Coupled – Sample analytical data, which includes the sample number (Client ID), are paired with the corresponding sample metadata. The Client ID is an important reference, as it is recorded in both the field notes and laboratory reports.

Dialysis Blanks – SPMDs received from the manufacturer and stored in a frozen state until shipped with environmental samples to the laboratory for chemical analysis. These blanks provide a measure of the concentration of organic compounds and performance reference compounds that are present in the SPMDs upon manufacture.

Deployed – Suspended in the water column for a predefined period of time.

Detection Limit – Limit below which a concentration may not be detected with any degree of confidence (see Sample-specific Reporting Limit and Method Detection Limit).

Dialysate – Extract from the dialysis of SPMDs.

Dialysis – Process of extracting compounds from SPMDs using a solvent.

Environmental Sample – An SPMD sample that has been used to measure ambient environmental conditions (i.e., water sample, field blank).

ETI – estimated time-integrated dissolved concentration, as calculated from

ng/sample concentrations using the USGS Estimator.

Extract – The product of SPMD dialysis completed in the laboratory. Extracts are concentrated prior to chemical analysis, which generates a concentration of compounds that accumulate in the SPMD membranes.

Field Blanks – SPMDs that are opened in the field and exposed to the air during deployment and retrieval of the water samples. These blanks accompany the water samples from manufacture through storage to chemical analysis, except during in-water deployment.

Flag – Non-numeric code assigned to a measured analyte concentration, identifying deficiency or bias in the data value.

Initialization – Entry of sample metadata into a database prior to sample collection and/or analytical data upload.

Lot Number – SPMDs are manufactured in lots (batches), which may vary in chemical composition.

Management (data) – Procedures relating to data from the moment of receipt from the laboratory to the storage of quality-assured data in a database.

Measure (sample, data) – Individual analyte concentration, as measured in a laboratory.

Metadata – Sample-specific information, including site name and code, sample number, and sampling data and time.

Method Detection Limit (MDL) – The MDL is defined herein as the limit below which an analyte concentration is considered indistinguishable from the analytical

background. The MDL is equal to the mean plus three times the standard deviation of analyte concentrations in laboratory blanks. Method detection limits are also determined by the laboratory using standard methods.

Nanograms per Litre (ng/L) – Estimated, time-weighted, dissolved concentration.

Nanograms per SPMD (ng/SPMD) – Raw concentration in a sample, as measured in the laboratory.

Non-detect – Measurement of an analyte, as reported by a laboratory, that is identified as below the analytical limit of detection (see “Sample-specific Reporting Limit”) and qualified with a “<”.

Octanol : Water Partitioning Coefficient (logKow) – Estimate of the solubility of a compound, expressed as the proportional partitioning of the compound between lipids (i.e., surrogate for biological organisms) and water.

Outlier – Analyte concentration that lies above or below a predefined limit (e.g., three times greater than the maximum of the main scatter body) and, which upon investigation, is shown to be erroneous and warrants qualification (e.g., RangeH).

Performance Reference Compounds – Compounds spiked into the SPMDs upon manufacture. These spikes are not naturally occurring and are used to calibrate the concentrations of compounds measured in the SPMD water samples for the purposes of converting the data to estimated dissolved concentrations.

Qualified / Qualification (data) – Data that have been assigned a non-numeric code (“flag”) to denote data that are biased or deficient, or to provide other essential information about the data.

Quality Assurance (data) – Identifies the requirements and procedures for the

generation of quality-assured and quality-controlled data. This includes program design, methods, and objectives.

Quality Control (data) – Consists of procedures and routine checks to ensure that the data are complete, correct, and consistent. Quality control includes approaches to data acquisition (field, lab) and handling, use of standard operating procedures, collection of replicate and blank samples, and data verification and validation.

Raw Data – Data received from the laboratory. These data are reported in ng/sample.

Recovery – A measure of the percent of a spike that has been recovered following chemical analysis in the laboratory.

Retrieved – Recovered from the water column after a predefined period of deployment.

Sample (type) – An SPMD sample consists of three membranes (see “Composite Sample”). SPMD sample types include environmental samples (i.e., “water samples” and “field blanks”, defined herein) and blanks (travel, dialysis, and spike blanks).

Sample-specific Reporting Limit (RL) –The RL, which is also known as the sample detection limit, may be defined as the limit below which a sample-specific analyte concentration may not be detected with any degree of confidence. The RL accounts for matrix effects on detection and recovery. This value is determined for each sample by the laboratory; the RL is calculated by converting the area equivalent of three times the estimated chromatographic noise height to a specific concentration (AXYS Analytical Services Ltd.).

Spike – A compound that is added to media or extracts in a known quantity to measure, and therefore correct for, its loss (see “Surrogate”, “Performance Reference Compound”, “Recovery”).

Spike Blanks – SPMDs that have not been spiked with performance reference compounds. Upon receipt from the manufacturer, these blanks are stored in a frozen state until shipped with environmental samples to the laboratory for chemical analysis. These blanks provide a measure of the concentration of organic compounds that are present in the SPMDs upon manufacture prior to spiking.

Statement of Work (SOW) – A written description of the scope and requirements of the work that is to be completed by a contractor. This will include quality assurance/quality control requirements.

Surrogate – A (standard) deuterated compound that is added, in a known quantity, to a sample in the laboratory prior to processing and analysis, to measure its loss. Measures of analytes that are of similar chemical characteristics to the surrogate may be corrected by taking the loss of this surrogate into account.

Travel Blanks – SPMDs that remain sealed in their tins and accompany the water samples and field blanks for the life of the samples (from manufacture through storage to chemical analysis).

Treatment (data) – Procedures for the background correction and conversion of raw data (ng/SPMD) to estimated time-integrated dissolved concentrations (ng/L).

Validation – Checks for the scientific validity of the data that may result in qualification of the data at a sample or measurement level. These checks may be based upon, where available, historical and/or expected ranges of data values.

Verification – Checks for data completeness, accuracy, and consistency.

VMV Code – Valid method variable code. This code is unique to each parameter and the analytical method applied for its measurement. The code is assigned by

Environment and Climate Change Canada (ECCC) and agreed to by the laboratory.

Water Samples – SPMDs that are deployed in the water for a predefined period of time.

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1.0 Introduction

The following standard operating procedures (SOPs) are intended for use on data generated from passive water quality monitoring using semi-permeable membrane devices (SPMDs). Where applicable, the data are handled in a manner consistent with national Environment and Climate Change Canada (ECCC) protocols.

These procedures are designed to support the production of data that are relevant, accurate, timely, accessible, interpretable, and coherent (Statistics Canada 2014). These are the basis for the ECCC Freshwater Quality Monitoring and Surveillance (FWQMS) Quality Assurance Framework. The framework aims to assure that ECCC's Freshwater Quality Monitoring Program data meet common quality standards across Canada (ECCC 2013).

Standardized procedures for the management, quality assurance (QA), quality control (QC), and treatment of SPMD data have not been published to date. Guidance provided by the United States Geological Survey (USGS 2010) and approaches referred to in the literature (Seiders and Sandvik 2013) were taken into consideration in the development of the approaches described herein.

1.1 SPMD Devices and Data Types

Semi-permeable membrane devices are membranes that are used to measure passively accumulated polycyclic aromatic compounds (PACs). Each membrane is composed of lay-flat, low-density polyethylene tubing that contains a thin layer of a pure, high-molecular-weight lipid (triolein). One sample consists of a composite of three membranes. The sample types and their applications are listed in Table 1.

The SPMDs are manufactured by Environmental Sampling Technologies (EST http://www.est-lab.com/). The concentration content of PACs in SPMDs, as measured in laboratory, are represented by the total concentrations of compounds that are present in the SPMDs at the time of chemical analysis. These compounds may originate not only from the waters that were sampled, but also from a number of other sources. These sources may include: i) membrane manufacture; ii) membrane exposure to air in the field (including transport and storage); and iii) membrane and extract handling and chemical analysis in the laboratory. Compounds in these instances are considered "background" and are quantified from quality control samples (i.e., dialysis blanks, field blanks, travel blanks, and laboratory blanks; Figure 1).

All membranes, except for the spike blank, are spiked by EST with 10 µg of fluoranthene-d10 and 10 µg of anthracene-d10. These performance reference compounds (PRCs) are used to calibrate for differences in conditions (e.g., temperature, membrane biofouling, and water/air flow at the membrane surface; Huckins et al. 2002) between sites and over time, and they are also used to calculate the estimated time-integrated (ETI), dissolved concentrations of organic compounds in the water column. The PRCs do not occur naturally and may be lost

during transport or storage, exposure to air in the field, and laboratory handling and chemical analysis (Figure 1). Concentrations of these compounds in dialysis blanks, spiked into the membranes upon manufacture, vary between manufacture lots, as well as between membranes ordered at different times (ECCC unpublished data). Initial and final PRC concentrations define the rate of exchange of PACs between the water column and the device (see Section 5.3).

Table 1 – Semi-permeable membrane device types and applications

Sample Type	Handling	Application
Water Sample	Deployed in the water.	Measuring levels of dissolved compounds in the water column. Measuring final PRC concentrations resulting from sample deployment in water.
Field Blank	Transported with the water sample and opened during field deployment and retrieval.	Measuring exposure of the water sample to the air, precipitation, and other sources of compounds during deployment and retrieval. Measuring initial PRC concentrations resulting from sample exposure to the air. One field blank accompanies each water sample. The field blank is used to define background concentrations for its companion water sample and to identify initial PRC concentrations that are used to calculate PAC exchange rates (i.e., in the USGS Estimator).
Travel Blank	Remains sealed and is transported with the water sample and field blank.	Measuring the exposure of the water sample and field blank during transport. Each QC sampling event (i.e., approximately 10% of sampling events) includes the collection of triplicate water samples accompanied by one field blank and one travel blank. The travel blank is used in data validation and assessment of program performance.
Dialysis Blank	Remains sealed and is stored in the freezer; shipped for analysis with other samples from the same lot.	Measuring background concentrations of compounds upon manufacture. Dialysis blanks for each SPMD lot (i.e., a minimum of three per batch/lot from the manufacturer). The dialysis blanks are used in data validation and in the assessment of program performance.
Day-zero Blanks (Spike Blanks)	Remains sealed and stored in the freezer; shipped for analysis with other samples from the same lot.	Measuring background levels of compounds upon manufacture. Not spiked with PRCs. Spike blanks for each SPMD lot (i.e., a minimum of three per batch/lot from the manufacturer). The spike blanks are used in data validation and the assessment of program performance.

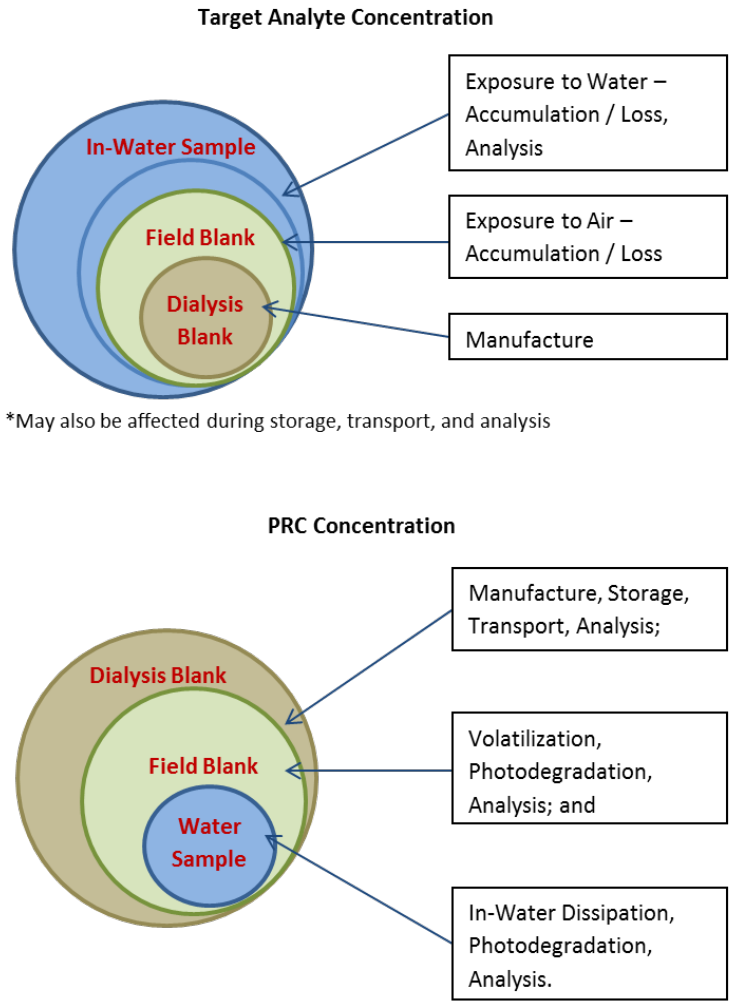


Figure 1. Target analyte and PRC concentrations may be measured using dialysis and field blanks that are companion to water samples.

Four types of data are generated from the ECCC passive water quality sampling program.

- **Sample metadata:** Sample-specific information, including site name and code, sample number, sampling date and time, and sample type (Table 1; e.g., water sample, field blank, travel blank, and dialysis blank).

- **Field data - Continuous:** Continuous water temperature data collected in situ using temperature loggers that are deployed with SPMDs and remain with the water samples until they undergo chemical analysis.

- **Field data - Observed:** Observations, site conditions, and water quality measurements collected in situ during the deployment and retrieval of SPMDs.

- **Analytical data:** Concentrations (ng/sample) of target analytes and PRCs in water samples and blanks (dialysis, field, travel, and laboratory), and associated QC information.

1.2 Data QA/QC

Standardized, quality-assured data results from the decisions and actions taken from program conception through long-term data storage. These include:

- **Planning:**
    - Definition of data requirements and objectives, analytical requirements, and QA process.
- **Implementation:**
    - Application of SOPs in the collection of samples and associated environmental information.
    - Collection of a predefined suite of QC samples.
    - Application of standard analytical methods and QC measures.
- **Verification and validation (addressed herein):**
    - Systematic verification of data completeness, accuracy, and consistency.
    - Assessment of data scientific validity and qualification of data as required.
- **Data treatment (addressed herein):**
    - Application of standard procedures for background-correcting and converting data.
    - Verifying and validating treated and converted data.
- **Storage:**
    - Application of standard procedures to upload the data to the database.
    - Secure storage and regular backup of data and associated records.
    - Regular updates to stored data, as needed, and records of changes made.

Passive water quality monitoring is QC intensive and generates large volumes of data and information relative to conventional water quality monitoring (Seiders and

Sandvik 2013). The collection of consistent, complete, and rigorously verified information is critical to data QA/QC, interpretation and reporting, and program performance assessment. Management to ensure the integrity and quality of the data should be based upon principles that include:

- **Timeliness of the data:** Ensuring that the data are acquired, up to date, and managed in a timely manner.

- **Quality assuring the data:** Ensuring that the data and associated treatments are complete and correct (i.e., supported by appropriate verification and validation procedures) and qualified (i.e., flagged) as necessary.

- **Tracking and storing the data:** Ensuring that data are maintained, complete, uploaded to a centralized data storage platform, and backed up.

1.3 Data Verification and Validation

Data verification and validation are necessary for the generation of quality data. Data verification consists of a series of checks for data completeness, accuracy, and consistency. The large volume of data associated with a passive water quality monitoring program requires that the data be managed closely at every step, from labelling and tracking samples, to verifying that the metadata, field data, and laboratory data meet quality requirements.

Data validation involves assessing the scientific validity of the data and qualifying samples and measures that are biased or deficient. Field and laboratory comments are important tools in the validation process. Samples are assessed to determine if they have been compromised. The analyte concentrations are validated to identify and assess concentrations that are outside the expected range, supporting the qualification of biased or erroneous measures.

1.4 Data Treatment

Semipermeable membrane devices are deployed in water bodies to measure target compounds or analytes that originate from the water. Data returned from the laboratory must undergo treatment to identify PAC concentrations that are attributable to the water sampled, and which may then be converted to ETI dissolved concentrations.

Treatment of SPMD data involves:

- pairing water samples and field blanks;

- background correction of verified and validated water sample concentrations; followed by

- conversion of raw ng/SPMD data into ETI dissolved ng/L.

Background correction has commonly been applied to SPMD data by subtracting the concentrations of analytes in a blank (or the mean of a set of blanks) from those in the environmental (water) sample (Wang et al. 2014, Sandvik and Seiders 2012, Helm et al. 2012, Era-Miller and Coots 2010). The resulting "net" concentrations of analytes are used to estimate dissolved concentrations.

The QC samples collected as part of the SPMD program include sample-specific field blanks, which represent the conditions upon manufacture, as well as the exposure of the water samples to the air during deployment and retrieval activities. To exclude potential site-specific extremes in analyte concentrations (in the air) from the water samples, the latter are background-corrected using their companion field blanks.

Concentrations of compounds accumulated in SPMDs deployed in water bodies are converted to ETI dissolved concentrations using the USGS Water Concentration Estimator. These estimated concentrations, which are calculated using the number of days deployed, initial PRC concentrations (as determined by field blanks), and final PRC concentrations (as indicated by the water samples), are calibrated to site conditions and therefore may be compared between sites and over time.

2.0 Sample Metadata

The management and QA of large volumes of data and information generated in a passive sampling program begins with meticulous allocation, tracking, and verification of sample metadata. Five types of samples are recommended for use in a water quality monitoring program that employs SPMDs (Table 1).

Upon completion of the program design and once the samples from the manufacturer have been received, each sample must be labelled with the following information:

- sample number;
- site code;
- site name;
- lot number (of the SPMDs received from the manufacturer); and,
- sample type (sample, field blank, travel blank, dialysis blank, or spike blank).

This sample metadata must be stored in an electronic sample file, which will become a repository for sample and field information (see Section 4.0, "Analytical Data"). This sample-tracking file must be consistently formatted, be updated

regularly, and be verified before the samples are shipped to the laboratory for chemical analysis.

The sample-tracking file will be used to:

- track sample status;
- record field data;
- create sample chains of custody (COCs);
- support verification of data delivered from the laboratory (see Section 4.0, “Analytical Data”);
- support data treatment (see Section 5.0, “Data Treatment”); and,
- initialize samples and enter field data into the database.

Chains-of-custody must be created and accompany samples when they are transferred between custodians. Samples that are shipped for chemical analysis are accompanied by a COC that must be populated with the required information, as specified by the laboratory (e.g., sample numbers, sampling date, or analytical suite – e.g., Appendix A). Upon completion and prior to shipping, the COCs should be checked carefully to ensure that:

- the sample metadata are correct and consistent with the verified data-tracking file;
- the requested analyses are specified correctly;
- the shipping custodian’s name and return address are specified; and,
- other pertinent information on the COC is provided.

Samples that are shipped to other ECCC offices must also be accompanied by a COC (see Appendix A) that includes the following information:

- sample number;
- site code;
- sample type;
- receiver name, address, and signature; and,
- the condition of the SPMD canisters upon receipt.

All samples are to be **identified by their retrieval date** when submitted for chemical analysis.

It is highly recommended that sample labels, data tracking files, and COCs be verified by an individual that has not been involved in the creation of these documents. The sample metadata must be consistent over the life of the sample (i.e., from labelling through to COCs to initialization in the database), minimizing the potential for errors in the sample numbers.

3.0 Field Data

Field data are collected during deployment and retrieval of the SPMDs following ECCC field SOPs (ECCC 2016). Data that are recorded on field sheets must be entered into the sample tracking file (see Section 2.0, “Sample Metadata”) and verified therein. These data records include:

- deployment date;
- retrieval date;
- coordinates (latitude and longitude);
- water depth (at deployment and retrieval);
- flow rate (if possible, at deployment and retrieval);
- physical water quality measures (water temperature, pH, dissolved oxygen, and turbidity and conductivity at deployment and retrieval);
- duration of exposure to air (for field blanks/samples during deployment and retrieval activities);
- weather conditions (e.g., clear skies, rain);
- comments and observations (e.g., damage to membranes, biofouling, bed sediment exposure, shipping concerns); and,
- the number of membranes per sample (i.e., three membranes equals one composite sample; if one membrane is damaged, it may not be included in the sample, reducing the number of membranes relative to the other samples).

The water temperature data loggers that accompany the water samples record water temperatures over the life of the sample (i.e., starting upon receipt from the manufacturer through to analysis of the retrieved sample). The temperature data are used to determine whether the sample was exposed to temperatures above the recommended maximum of 4°C (USGS 2010) and if the sample was removed from the water during deployment (thus compromising the sample). Following

sample retrieval and prior to sample submission for chemical analysis, it is important to:

- download the logger data;
- calculate the mean, minimum, and maximum temperatures for each logger; and,
- identify samples that have been exposed to air during their deployment in the water; these samples will not be submitted for analysis.

These field data and sample metadata are used to support data QA/QC, treatment, and conversion. This information is also valuable to program performance assessments, as well as data analysis, interpretation, and reporting. As such, it is important to ensure that the data are recorded in a standard format and that the records are complete, up to date, and secure. Field data verification and validation are critical to generating quality ETI dissolved concentration data.

3.1 Verification

The field data are verified to ensure that the information entered into the electronic sample tracking file is consistent with that in the field sheets. Verification can also promote the identification and correction of errors in the field sheets while ensuring that quality-assured data are available for entry into the database.

Within the sample tracking file:

- verify the sample metadata (Section 2.0, “Sample Metadata”);
- verify the field data (Section 3.0, “Field Data”);
- revise as necessary and record the rationale for revisions; and,
- record the name of the individual who completed the verification and document the date it was completed.

Verification should not be completed by the individual who entered the data into the tracking file. Verification of the field data should ideally be completed by the individual who collected the sample or by someone who has knowledge of the program design, sites, and expected conditions (e.g., someone who can identify what may constitute an erroneous value).

3.2 Validation

Field records are used to screen samples for chemical analysis and to qualify the sample. Complete the following checks to determine if a sample has been compromised or biased.

- **Temperature logger data:**

    - Has the water sample been exposed to the air during the period of deployment, as indicated from the temperature logger record? If so, this sample will not be submitted for chemical analysis.

    - Has the water sample (during storage and/or transport) been exposed to temperatures that far exceed the recommended threshold of 4°C? If so, the water quality monitoring program lead may elect to qualify the sample.

- **Sediment exposure observations:** Has the water sample been exposed to bed sediments, as observed in the field and indicated on the field sheet? If so, this sample will not be submitted for chemical analysis. Alternatively, the program lead may elect to qualify the sample.

- **Air exposure times:** Has the water sample been exposed to the air for an excessive period of time during the retrieval process? If so, the program lead may elect to qualify the sample.

- **Field comments:** Has the sample been definitively compromised or biased (e.g., lid of the tin not sealed, membrane damaged/ripped, or sample exposed to heavy rainfall on retrieval)? If so, the program lead will qualify the sample.

A sample qualified through these checks will be assigned an “EVENTF” flag that will be applicable to all measurements in the sample (Section 4.0, “Analytical Data”).

- ❖ **Exception:** If the laboratory indicates that a sample is compromised due to field conditions that were not identified on the field sheets (e.g., the membrane is coated with a tar-like substance that could not be removed prior to dialysis) and that this will affect the target analyte concentrations, this sample and its associated measures must be qualified as “EVENTF”.

"EVENTF" qualifies a sample (i.e., all measurements) that is determined to be biased or compromised.

Applies to:

ng/SPMD data (all sample types)

ng/L data (water samples)

4.0 Analytical Data

Samples are submitted for chemical analysis to determine concentrations of parent and alkylated PACs that accumulated in the SPMDs. Quality assurance of these data begins with selection of a laboratory that will meet the data objectives and QA/QC requirements of the water quality monitoring program. These requirements, the details of which will be specified in analytical contracts, will include rigorous and standard analytical and QC methods, clear and comprehensive reporting, and timely and complete data delivery.

Analytical data must be verified to ensure that the data are correct. This will include verification of the delivered data and the sample metadata; the latter must be coupled to the analytical data prior to data validation and treatment.

4.1 Delivery Verification

The first step toward ensuring the quality of data received from a laboratory includes performing a thorough review of the data against predefined requirements of the data. These requirements will be defined by the monitoring program and the program lead. If the analytical work was contracted, these requirements must be contract specific (e.g., established in a statement of work).

A thorough review of the delivered data will ensure that program and, if applicable, contract requirements are met. The review process, as well as uploading of data to a database, will be facilitated and errors will be minimized with the use of a predefined data format that is consistent and amenable to upload. The data should be delivered electronically in the predefined format, and they should also be summarized and presented along with analytical QA/QC information, including case narratives, in a written report.

Electronic data files (see Appendix B) must be reviewed and include the following at minimum:

- a laboratory code (a unique code provided by the laboratory);
- a laboratory sample number;

- an ECCC sample number;
- a sample date (retrieval date);
- a sample time;
- a method code (e.g., a valid method variable [VMV] code);
- a detection limit (sample-specific reporting limit [RL] or, in some cases, the method detection limit [MDL]);
- raw data (analyte concentrations in ng/sample and surrogate recovery results in percent to monitor program samples and laboratory batch-specific QC samples);
- data qualifiers (assigned by the laboratory, indicating that a measure does not meet laboratory QA criteria); and,
- the number of membranes per sample.

In addition, written reports must include:

- a description of methods (dialysis, cleanup, chemical analysis, and quantification); and,
- a case narrative (i.e., a record of sample-specific analytical issues or mishaps, and a description of the resulting bias in data measures).

Verification of deliverables should be completed or directed by the program lead that established the requirements. It is critical that the verification be completed in a timely manner; if errors are found, the laboratory may have to investigate the data or re-analyze the samples, causing delayed or impeded data delivery.

The series of checks may include verification of data format and content, written report content, detection limit requirements, and QA/QC measures and results, including surrogate use and recovery. Table 2 lists examples of questions that may be used to verify deliverables.

Table 2 – Deliverable verification

Step	Question
Receiving	Has the contractor confirmed receipt of all samples and reported the temperature upon arrival?
	Has the contractor returned all shipping and sample materials?
Reporting	Were the data provided in the required format?
	Do the data include all required samples?
	Do the data include all required analyte measures?
	Are data qualifiers defined?
	Are measures that are less than the detection limit reported as required?
Analysis	Have the required detection limits been met?
	Have all required surrogates been used and their recovery reported?
	Have performance reference compounds been avoided as surrogates?
	Are surrogate recoveries within acceptable limits?
Quality Control	Do the data include i) a method/laboratory blank, and ii) a laboratory control/method spiked matrix for each batch?
	Are analyte measures in the laboratory blanks below their detection limits?
	Are recoveries of analytes in the method spike within acceptable limits?
	Does the written report contain the required quality control information?

4.2 Metadata Verification

Once the deliverables and the contract criteria have been verified, the samples and associated analyte data must be coupled with the field metadata. This will be automated when uploading the data to a database, which is where the samples and field information have been initialized. The sample identification (YYYYPN##****) assigned for the monitoring program is key in bringing together the analytical data, sample metadata, and field observations (see Section 3.0, “Field Data”).

The coupling of analytical data with the appropriate metadata is a critical step in ensuring data quality. The ability to differentiate between sample types (e.g., field blank versus dialysis blank) and to pair samples with their companion blanks (e.g., water samples and field blanks) must exist in order to validate, treat, and convert the data to ETI dissolved concentrations.

Couple the following metadata to the analytical data (following detailed instructions in the Excel template, where applicable):

- site name;
- sample type (e.g., water sample, blank type, and triplicate);
- start date (deployment date);

- end date (retrieval date);
- lot number (i.e., where more than one manufactured lot of membranes is used);
- number of membranes; and,
- field comments.

Within the file (following detailed instructions in Excel template, where applicable):

- verify the coupling of the sample metadata;
- verify the integrity of the raw data (analyte concentrations);
- examine PRC concentrations to ensure they are consistent with the sample type (e.g., water sample < blanks); and,
- record the name of the individual who completed the verification and the date it was completed.

4.3 Data Validation

Analytical results are validated to identify whether they are scientifically sound or if they should be assigned qualifiers. Data validation, which will be partly based on scientific judgement, should be completed by the program lead. Qualifiers will be assigned to the data if a sample or measure is found to be deficient, biased, or within background. Data that are deemed erroneous will be investigated and corrected as required.

4.3.1 Laboratory QC Information

The laboratory reports QC results and qualifies data when laboratory quality criteria are not met. This information is used to assess performance and implement corrective measures, if necessary; assess data limitations and take these into account in data validation and interpretation; and identify samples and/or measures that are sufficiently biased concentrations that spark concern during data interpretation.

Following verification of the deliverables received from the laboratory:

- review the data for laboratory qualifiers that indicate erroneous results;
- review the case narratives to determine if the sample and associated results are biased or have been compromised
    - if so, and the cause occurred in the laboratory, qualify the sample with “EVENTL”; and,

- if so, and the cause occurred in the field (e.g., residue on the membrane was included in the analysis or the membrane was damaged upon arrival at laboratory), qualify the samples with “EVENTF”.

“EVENTL” qualifies a sample (i.e., all measurements) that is determined to be biased or compromised. Applies to: ng/SPMD data (all sample types) ng/L data (water samples)

Examples of laboratory QC information associated with SPMD data are provided in Appendix C.

4.3.2 Range of Concentrations

Raw data are examined for extreme values (“outliers”) that may result from error or atypical conditions (e.g., transcription error, or field or laboratory incidents). Tools to identify outliers include scatterplots, box plots, confidence intervals, and thresholds based on historical values or a predefined order of magnitude difference from other data points (Ohio EPA 2012). The ECCC passive sampling dataset is currently insufficient for establishing statistical or historical thresholds.

Following the receipt of sample data from the laboratory, deliverable verification, and metadata coupling, and prior to data treatment:

- gather the raw data from, for example, one monitoring year in a select monitoring region;
- create a scatter plot for i) each analyte in the water samples, and ii) each analyte in blanks (grouped by blank type);
- examine the scatterplots for outliers (those values that are sufficiently extreme and distinct in magnitude from the main body of the scatter points (i.e., three times greater than the maximum of the main scatter body; Figure 2); and,
- proceed with an assessment of the outliers:
    - if a concentration is atypically high and deemed biased or erroneous, qualify the measurement with “RANGEH”,
    - if a concentration is atypically high and deemed biased or erroneous, qualify the measurement with “RANGEL”.

“RANGEH” or “RANGEL” qualifies an extreme measurement that is determined to be biased or erroneous.

Applies to:

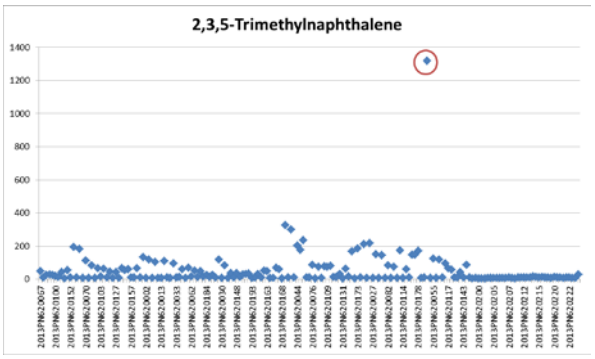
ng/SPMD data (all sample types)

ng/L data (water samples)

The decision to qualify outliers should be made by the program lead. The assessment of outliers for qualification is based on:

- frequency (e.g., “extreme” values that occur in the same sample across multiple and/or similar analytes may be the result of a natural event; however, field or laboratory comments may indicate contamination as the source);
- detection in both the sample and companion field blank (this suggests that the sample has not been compromised);
- field comments (this will indicate if the sample was likely compromised in the field);
- field conditions (e.g., high turbidity and/or conductivity, which would indicate that [multiple] compound concentrations will be elevated);
- laboratory case narrative (this will indicate whether the sample was likely compromised in the lab); and,
- laboratory data qualifiers (may help to inform the interpretation of extreme values).

a)



b)

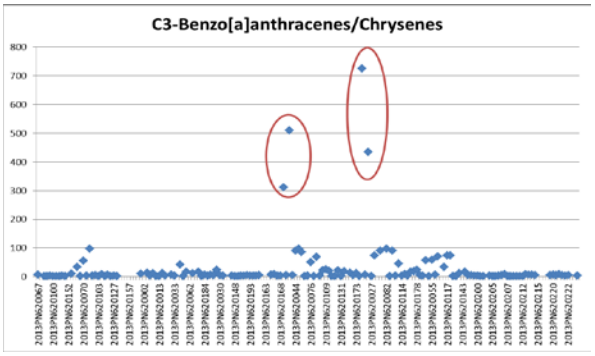


Figure 2. Example of extreme values associated with a) high turbidity (do not qualify) and b) a broken seal on the tin (qualify).

The possible cases and causes of outliers include:

- concentrations of analytes in QC blanks (field, travel, or dialysis) that are equivalent to or higher than those in the water samples (indicating potential contamination of the water sample);
- concentrations of analytes in travel blanks that are higher than those in the field blanks and/or dialysis blanks (indicating a need to investigate travel conditions and practices);
- concentrations of analytes that are dissimilar between replicate samples (indicating the potential compromise of one replicate sample);
- concentrations of analytes that are higher in the laboratory blanks than in the water quality program QC blanks (indicating the need to consider laboratory conditions and performance); and,
- concentrations of PRCs that are not consistent with the sample type (indicating potential mislabelling of samples).

Outliers that are clearly deemed erroneous or biased, and which are attributable to events that have occurred in the laboratory (Section 4.3.1, “Laboratory QC Information”) or in the field (Section 3.2, “Validation”), that have compromised the sample must be qualified with a “RANGEH” or “RANGEL” flag. Extreme values

that are associated with high turbidity and extreme flow, for instance, will not be qualified (e.g., Figure 2b).

5.0 Data Treatment

Data treatment is the process of pairing, background correcting, and converting PAC concentrations in water samples to ETI dissolved concentrations. Procedures for doing so have not been standardized (Era-miller and Coots 2010). The following data treatments are applied to the raw data:

- replacement of measurements identified as non-detects (below the analytical limit of detection) with the sample-specific reporting limit;
- replication of field blanks;
- background correction of water sample concentrations; and,
- conversion of ng/SPMD concentrations to ng/L.

5.1 Pairing

Prior to pairing and background correcting the water samples, both water samples and field blanks must have non-detect measurements replaced with their respective sample-specific RLs (RLs). These measurements are identified with a "<" symbol, indicating that they are less than the analytical detection.

After coupling the raw data to the sample metadata and associated verification:

- replace all water sample non-detects with their RLs;
- replace all field blank non-detects with their RLs;
- verify the replacement; and,
- retain the "<" symbol as a flag for these measures.

The "<" symbol indicates that a measurement is below the analytical detection limit.

Applies to:

ng/SPMD data (all sample types)

ng/L data (water samples)

The water samples must be paired with their companion field blanks before their concentrations can be background corrected and converted. To pair samples:

- assign a common and unique identifier to each water sample and associated field blank;
- where replicate water samples exist, replicate data from companion field blanks (including associated unique identifiers), such that all water samples have companion field blank data;
- create two spreadsheets that are identical, except for the concentrations used (i.e., one for water samples, one for field blanks); and,
- verify the pairing and field blank replication.

5.2 Background Correction

Analyte concentrations in each water sample are background corrected by subtracting the concentrations of the analytes in the companion field blank from those in the water sample. Following verification of the sample pairings and replication:

- subtract the field blank concentrations from their companion water samples;
- retain the positive net concentrations;
- replace the negative net concentrations with the corresponding water RLs:
    - assign these measures a “BLANKF” flag; and,
- verify the subtraction and flagging.

“BLANKF” qualifies a sample measurement that is lower than that in the corresponding field blank. Applies to: ng/SPMD data (all sample types) ng/L data (water samples)

These net values are converted to ETI dissolved concentrations.

5.3 Performance Reference Compounds

Initial and final concentrations of PRCs are required to calibrate and convert the ng/SPMD concentrations to ng/L. Initial concentrations are defined as the median concentration within the field blanks for a given manufacture lot in a given month; final concentrations are those that are measured in the water samples subsequent to retrieval.

Using the paired water samples and field blanks (Section 5.2, “Background Correction”):

- identify the final PRC concentrations for each water sample;
- where final concentrations are below the limit of analytical detection (i.e., non-detectable), assign the RL;
- compile the field blanks from each month (for a predefined geographic region) and calculate the median concentration of each PRC;
- pair this median initial PRC concentration with all final PRC concentrations from water samples collected in that month; and,
- verify the calculation and pairing of PRC concentrations.

These PRC concentrations will be entered into the USGS Water Concentration Estimator.

5.4 Conversion

The USGS Water Concentration Estimator is run for each sample. The input for each sample is tailored to the number of days that the sample was in the water, the initial and final PRC concentrations, and the concentration of target analytes measured in the sample. The Estimator produces a concentration of target analytes in picograms per litre (pg/L).

Prior to running the Estimator, the following information must be compiled:

- analyte names (retaining the order of the names as for the net concentrations);
- preferred octanol:water (Log Kow) partitioning coefficients for the analytes (see Appendix D);
- for each sample, the total number of days the SPMDs were deployed (stored in the data tracking file; see Section 3.0, “Sample Metadata”);
- for each sample, the verified initial and final concentrations of PRCs; and,

- for each sample, the verified net analyte concentrations (ng/SPMD).

Once this information is compiled:

- Create an Estimator worksheet that:
  - contains the names of the analytes (as for the net concentrations), and
  - their partitioning coefficients.
- Replicate this worksheet for each sample.
- Assign each sheet a sample-specific name (following the preferred naming convention).
- Within each worksheet enter the sample-specific:
  - sample number and sample date;
  - total number days deployed;
  - initial and final PRC concentrations; and
  - net analyte concentrations.
- Verify these Estimator inputs.
- If the concentrations of a PRC are such that one must be excluded (e.g., the initial concentration is less than the final concentration), assign a “PRC” flag to all measures in the sample.
- Verify the Estimator output.

“PRC” qualifies a sample (i.e., all measurements) that does not have the standard set of PRCs. Applies to: ng/L data (water samples)

6.0 Data Qualifiers

Data qualification allows for data retention, while ensuring that the data user understands the limitations of the data. The processes of verifying, validating, and treating the field and laboratory data may result in qualification of the data at the sample or measurement level (Table 3). Data qualifiers are stored and released with the data. The QA process is summarized in Figure 3.

Table 3 – Scenarios for qualifying data

Flag	Example
EVENTF (sample)	Water sample exposed to bed sediments during deployment; if submitted for chemical analysis, the concentrations will not represent those in the water column.
	Water samples exposed to the air during deployment; if submitted for chemical analysis, the concentrations will not represent those in the water column.
	The sample has been compromised or damaged; if submitted for chemical analysis, the concentrations will not represent those in the water column.
	Due to field events, the sample was collected from a location downstream of the sampling site (and additional tributary or point sources); the concentrations do not represent those of the assigned sampling site.
	Due to field events, this sample was deployed for an abbreviated (less than 2 weeks) or extended (more than 6 weeks) period of time; the deployment period is inconsistent with that of the samples from the rest of the program.
	Field and laboratory information suggests that there was a coating that may compromise the films' ability to be sampled. Also, any residue from the coating would likely be included in the analysis.
	Laboratory information suggests that there is a tar-like substance on the film that would likely limit the sampling rate while in the field, and it would likely be included in the analysis.
EVENTL (sample)	Partial loss of dialysate. Analyte concentrations in the sample will be lowered.
	Abbreviated dialysis time. Analyte concentrations will be lowered.
	Due to a laboratory event, this sample was re-analyzed. The PAH concentration in the laboratory blank should be considered for this sample.
	Analyte concentrations in the laboratory blank for a batch of samples does not meet laboratory method blank control limits. The concentrations of the analyte in the samples from this batch may be biased.
EVENTL (sample)	Due to the "K" laboratory flag, the high concentration may be an overestimate.
	The phenanthrene parameters of the sample were flagged with "K" by the laboratory; therefore, the high concentration may be an overestimate.
	The can was open during the shipping process and it seems likely that the concentrations of volatile compounds were affected.
BLANKF (measurement)	The concentration in the field blank is higher than that in the companion water sample.
PRC (sample)	Start PRC concentration > end PRC concentration; therefore, the start concentration is deemed erroneous. Estimator values based on Fluoranthene d-10 OR Anthracene d-10 are PRC only.

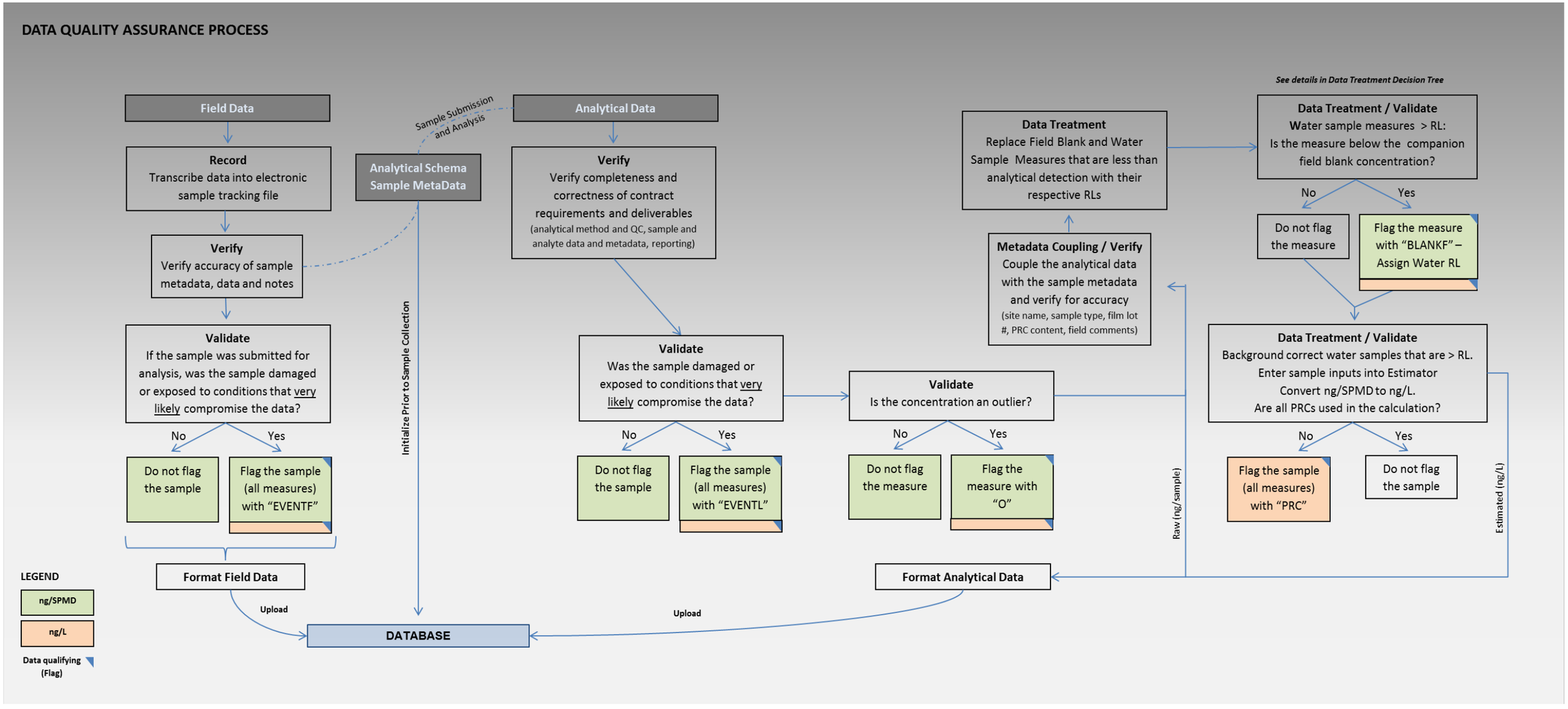


Figure 3. Quality assurance process.

7.0 References

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Appendix A. Chains of Custody

A-1. AXYS Analytical Laboratories COC

2045 Mills Road West TEL: (250) 655-5800
Sidney, British Columbia, Canada, V8L 5X2 FAX: (250) 655-5811

CHAIN OF CUSTODY

AXYS CLIENT #: 4540

REPORT TO:		*INVOICE TO:*	
Company	Environment Canada	Company	Environment Canada
Address	11 Innovation Blvd	Address	11 Innovation Blvd
	Saskatoon, SK		Saskatoon, SK
	S7N 3H5		S7N 3H5
Contact	Lucie Levesque	Contact	Lucie Levesque
Phone	306 975 6107	Phone	306 975 6107
FAX		FAX	
E-mail	lucie.levesque2@canada.ca cari-lyn.epp@canada.ca	E-mail	lucie.levesque2@canada.ca cari-lyn.epp@canada.ca
Project Name/Number: Project 349 WQMS SPMDs		Sampler's Name: Leah Dirk Signature:	

ANALYSIS REQUESTED

PAH LIST (1 TO 3)

Client Sample Identification	Matrix	Sampling Date	Sampling Time	Container Type/No.	AXYS Lab Sample ID (Lab use only)					
2016PN620001	SPMD	26-Jun-16	19:00	LG TIN		X				
2016PN620002	SPMD	26-Jun-16	19:05	MED TIN		X				
2016PN620004	SPMD	26-Jun-16	18:15	MED TIN		X				
2016PN620011	SPMD	29-Jun-16	12:30	LG TIN		X				
2016PN620012	SPMD	29-Jun-16	12:35	MED TIN		X				
2016PN620013	SPMD	29-Jun-16	12:40	SM TIN		X				
2016PN620014	SPMD	27-Jun-16	14:00	LG TIN		X				
2016PN620015	SPMD	27-Jun-16	14:05	MED TIN		X				
2016PN620016	SPMD	27-Jun-16	10:30	LG TIN		X				
2016PN620017	SPMD	27-Jun-16	10:35	MED TIN		X				
2016PN620018	SPMD	27-Jun-16	12:00	LG TIN		X				
2016PN620019	SPMD	27-Jun-16	12:05	MED TIN		X				
2016PN620022	SPMD	27-Jun-16	14:55	LG TIN		X				
2016PN620023	SPMD	27-Jun-16	15:00	MED TIN		X				
2016PN620026	SPMD	27-Jun-16	15:05	SM TIN		X				
2016PN620029	SPMD	30-Jun-16	12:50	LG TIN		X				
2016PN620030	SPMD	30-Jun-16	12:55	MED TIN		X				
2016PN620031	SPMD	30-Jun-16	13:00	SM TIN		X				
2016PN620032	SPMD	27-Jun-16	13:15	LG TIN		X				

A-2. ECCC Inter-Office COC

ENVIRONMENT CANADA - SPMD INTERNAL COC

Sample #	Site	Sample Type (Sample, FB, TB, DB)	Condition on Receipt Shipment #1	Condition on Receipt Shipment #2
2014PN620152	M9	Sample 10ug		
2014PN620153	M9	FB 10ug		
2014PN620154	M11a	Sample 10ug		
2014PN620155	M11a	FB 10ug		
2014PN620156	M12	Sample 10ug		
2014PN620157	M12	FB 10ug		
2014PN620158	BI1	Sample 10ug		
2014PN620159	BI1	FB 10ug		
2014PN620160	RI1	Sample 10ug		
2014PN620161	RI1	FB 10ug		
2014PN620162	QU1	Sample 10ug		
2014PN620163	QU1	FB 10ug		

SHIPMENT #1		
Shipped by:		**Received by:**
Name (printed)		Name (printed)
Signature		Signature
Location		Location
Date Shipped		Date Received
TOTAL Days in Transit:		

SHIPMENT #2		
Shipped by:		**Received by:**
Name (printed)		Name (printed)
Signature		Signature
Location		Location
Date Shipped		Date Received
TOTAL Days in Transit:		

Appendix B. Reporting

B-1. Database.xls File

LabCode	ClientID	SubmitterID	VMVcode	DetLim	Flag	Result	AnalysisDate	Station	SampleDate	SampleTime	TimeZone	AxysID	Method	ReceiptDate	Strips
135	2013PN620044		110092	2.06	J	30.4	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110031	2.95	KJ	6.99	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110030	17.2	KJ	25.6	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110020	25.3	KJ	29.2	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110053	65.0		4960	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110088	13.7	KJ	20.9	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110094	13.1		242	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110032	12.6	KJ	44.6	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110046	31.4		1920	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110087	43.6		172	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110095	41.5		956	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110033	25.3	KJ	41.3	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110065	29.2		860	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110037	7.61	K	61.0	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110038	8.00	KJ	20.5	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110035	10.9	K	107	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110034	11.4	J	24.1	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110093	11.6		377	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110084	3.43	KJ	3.47	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110089	5.74	KJ	9.27	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110036	4.82	J	20.2	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110021	3.80	J	24.0	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110011	4.06	J	16.3	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110045	3.80		40.3	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110039	3.92	J	4.26	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110091	2.61		7.50	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110086	2.64		62.4	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110052	9.97		163	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110005	9.97	KJ	25.1	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110016	8.52	KJ	18.9	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110058	29.2		998	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110014	28.0		163	2014.07.09		2013.08.21			L21633-30		2014.06.27	3
135	2013PN620044		110013	30.6	K	204	2014.07.09		2013.08.21			L21633-30		2014.06.27	3

B-2. DataSummary.xls File

CLIENT_ID	2013PN620044			2013PN620045			Lab Blank (101)			Spiked Matrix (102)		
Axys ID	L21633-30			L21633-31			WG47974-101			WG47974-102		
WORKGROUP	WG47974			WG47974			WG47974			WG47974		
Sample Size	1sample			1sample			1sample					
UNITS	flag	ng/sample	ng/sample (RL)	flag	ng/sample	ng/sample (RL)	flag	ng/sample	ng/sample (RL)	flag	% Recovery	-
Naphthalene	J	30.4	2.06	J	31.3	1.98	J	6.99	1.81		97.4	
Acenaphthylene	K J	6.99	2.95	K J	3.89	3.85	ND		0.905		96.2	
Acenaphthene	K J	25.6	17.2	K J	30.7	9.93	ND		1.39		99.4	
2-Methylfluorene	K J	29.2	25.3	K J	37.5	23.3	ND		1.39		94.1	
C2 Phenanthrenes/Anthracenes		4960	65		5030	27.9		1.26	0.666			
Fluorene	K J	20.9	13.7	K J	14.5	6.62	ND		0.658		88.5	
Phenanthrene		242	13.1		231	12.7	J	1.07	0.806		96.9	
Anthracene	K J	44.6	12.6	K	49	12.2	K J	1.54	0.775		91.5	
C1 Phenanthrenes/Anthracenes		1920	31.4		1900	32	ND		1.06			
Fluoranthene		172	43.6		183	47.2	J	0.684	0.522		107	
Pyrene		956	41.5		1030	45	J	0.643	0.498		103	
Benz[a]anthracene	K J	41.3	25.3	K J	39.6	14.2	J	0.754	0.435		99.2	
Chrysene		860	29.2		873	16.5	K J	0.457	0.44		100	
Benzo[b]fluoranthene	K	61	7.61		64.3	4.87	ND		1.03		98.8	
Benzo[j,k]fluoranthenes	K J	20.5	8	J	18.4	5.37	ND		1.11		99.5	
Benzo[e]pyrene	K	107	10.9	K	120	7.2	ND		1.55		105	
Benzo[a]pyrene	J	24.1	11.4	J	23.9	7.5	ND		1.61		101	
Perylene		377	11.6		384	7.63	ND		1.65		100	
Dibenz[a,h]anthracene	K J	3.47	3.43	K J	3.62	3.56	ND		1.73		98.3	
Indeno[1,2,3-cd]pyrene	K J	9.27	5.74	J	12.2	2.93	ND		1.94		102	
Benzo[ghi]perylene	J	20.2	4.82	J	20	2.48	ND		1.62		99.4	
2-Methylnaphthalene	J	24	3.8	J	26.9	1.9	J	2.02	1.61		99.3	
1-Methylnaphthalene	J	16.3	4.06	J	18	2.03	ND		1.72		99.2	
C1-Naphthalenes		40.3	3.8		44.9	1.9		2.02	1.61			
Biphenyl	J	4.26	3.92	J	5.19	3.03	J	2.33	1.3		97.6	
C1-Biphenyls		7.5	2.61		9.34	2.87		1.79	0.953			
C2-Biphenyls		62.4	2.64		66.1	5.97		7.32	0.903			
C2-Naphthalenes		163	9.97		209	9.66		5.47	2.48			
1,2-Dimethylnaphthalene	K J	25.1	9.97	K J	32.7	9.66	ND		2.48		98.1	
2,6-Dimethylnaphthalene	K J	18.9	8.52	K J	24.1	8.25	ND		2.12		98.9	
C3-Naphthalenes		998	29.2		1140	26.1		10.1	1.04			
2,3,6-Trimethylnaphthalene		163	28		161	25	ND		0.993		108	
2,3,5-Trimethylnaphthalene	K	204	30.6	K	176	27.3	ND		1.08		106	
C4-Naphthalenes		5340	136		4860	136		3.01	1.25			
C1-Acenaphthenes		15.4	3.96		14.2	6.62	ND		1.25			
C1-Fluorenes		396	25.3		429	23.3		5.16	1.39			
1,7-Dimethylfluorene	K	234	70.9		240	23.7	ND		1.64		114	
C2-Fluorenes		3220	70.9		3370	23.7		3.67	1.64			
C3-Fluorenes		5960	86.2		5840	181		6.79	2.92			
Dibenzothiophene	K J	31.4	26.6	ND		35.6	ND		0.697		99.2	
C1-Dibenzothiophenes		609	70.9		415	65.3	ND		1.61			
2/3-Methyldibenzothiophenes	K	246	70.9	K	246	65.3	ND		1.61		77.7	
C2-Dibenzothiophenes		8470	28.3		8630	90.6		2.55	2.03			
2,4-Dimethyldibenzothiophene	K	530	28.3	K	525	90.6	ND		2.03		74.6	
C3-Dibenzothiophenes		9880	64.7		9850	41.8		5.48	1.24			
C4-Dibenzothiophenes		3980	58.5		5270	57.2		18.1	2.03			
3-Methylphenanthrene		406	31.4		391	32	ND		1.05			
2-Methylphenanthrene		407	31.4		404	32	ND		1.05		104	
2-Methylanthracene	ND		31.4	ND		32	ND		1.06		94.2	
9/4-Methylphenanthrene		749	31.4		742	32	ND		1.05			
1-Methylphenanthrene		362	31.4		360	32	ND		1.06		107	
3,6-Dimethylphenanthrene	K	468	66.1	K	456	28.4	ND		0.678		104	
2,6-Dimethylphenanthrene	K	244	65	K	247	27.9	ND		0.666			
1,7-Dimethylphenanthrene		680	63.9		685	27.4	ND		0.655		106	
1,8-Dimethylphenanthrene		200	65		200	27.9	ND		0.666			
C3-Phenanthrenes/Anthracenes		5620	43.1		5450	49.9		2.05	0.895			
1,2,6-Trimethylphenanthrene		241	43.1	K	254	49.9	ND		0.895		104	
Retene		678	135		659	73.3	ND		1.59		104	
C4-Phenanthrenes/Anthracenes		8160	135		9590	73.3		7.8	1.59			
C1-Fluoranthenes/Pyrenes		5750	151		6270	159	ND		1.93			
3-Methylfluoranthene/Benzo[a]fluorene		936	151		1020	159	ND		1.93			
C2-Fluoranthenes/Pyrenes		5690	53.7		6100	53.1		2.74	1.07			
C3-Fluoranthenes/Pyrenes		1440	20.9		2280	18.9		2.89	1.18			
C4-Fluoranthenes/Pyrenes		501	16.8		643	16.7		1.89	1.07			
C1-Benzo[a]anthracenes/Chrysenes		906	9.07		936	6.2		1.53	0.56			
5/6-Methylchrysene		74	9.25		74.5	6.32	J	0.768	0.571		95.9	
1-Methylchrysene		98.5	8.9		103	6.08	J	0.618	0.55		95.6	
C2-Benzo[a]anthracenes/Chrysenes		535	3.57		489	4.18		1.2	0.616			
5,9-Dimethylchrysene		113	3.57		94.5	4.18	ND		0.616			
C3-Benzo[a]anthracenes/Chrysenes		91.3	3.01		97	5.55		1.41	0.711			
C4-Benzo[a]anthracenes/Chrysenes		25.5	3.35		21.2	3.2		2.78	0.948			
C1-Benzofluoranthenes/Benzopyrenes		186	5.08		183	4.71		4.24	2.68			
7-Methylbenzo[a]pyrene	J	13.3	5.08	J	13.5	4.71	ND		2.68		92.6	
C2-Benzofluoranthenes/Benzopyrenes		46.3	7.58		43.8	4.07		2.49	1.86			
1,4,6,7-Tetramethylnaphthalene	K	1200	136	K	1170	136	ND		1.25		111	
Fluoranthene d-10		12000	113		11400	96.2		7.37	0.881		98.3	
Dibenzo[a,h]anthracene d-14	ND		4.1	ND		1.61	ND		2.3		88	
Anthracene d-10		2240	26.5		2070	42.5	ND		4.12		89.3	
Naphthalene d-8 (% Recovery)		71			61.5			46.2			57.9	
2-Methylnaphthalene d-10 (% Recovery)		77.8			70.5			47.6			61	
Biphenyl d-10 (% Recovery)		81.8			75.2			49.2			65.5	
2,6-Dimethylnaphthalene d-12 (% Recovery)		81.3			75.3			48.4			65.6	
Acenaphthylene d-8 (% Recovery)		85.2			79.3			49.4			66.8	
Dibenzothiophene d-8 (% Recovery)		83			75.3			37.4			57.4	
Phenanthrene d-10 (% Recovery)		91.8			81			65.4			85.6	
Benzo[a]anthracene d-12 (% Recovery)		84.5			81.8			76.7			81.5	
Chrysene d-12 (% Recovery)		81.3			79.1			78.8			82.5	
Benzo[b]fluoranthene d-12 (% Recovery)		94			86.6			85.1			93.7	
Benzo[k]fluoranthene d-12 (% Recovery)		90.8			82.7			87.7			94.8	
Benzo[a]pyrene d-12 (% Recovery)		86			77.2			79.4			87.7	
Perylene d-12 (% Recovery)		86.3			77.7			79.8			86.4	
Indeno[1,2,3-cd]pyrene d-12 (% Recovery)		72.3			57.3			69.4			71.2	
Benzo[ghi]perylene d-12 (% Recovery)		72.1			58.9			68.4			73.4	

B-3. PDF Report

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The AXYS contact for these data is Jenny Pape.

PAH ANALYSIS

SPMD SAMPLES

PROJECT NAME: PROJECT 349 OIL SANDS PASSIVE SPMD

Contract: 4540
Data Package Identification: DPWG48398

Analysis WG47974

19 August 2014

ENVIRONMENT CANADA
SPMD Samples

POLYCYCLIC AROMATIC HYDROCARBON ANALYSIS
AXYS METHOD: MLA-021
4540: L21633-30, -31, L21634-34 to -45, L21636-5 to -8

PROJECT NAME: PROJECT 349 OIL SANDS PASSIVE SPMD

19 August 2014

NARRATIVE

This narrative describes the analysis of eighteen SPMD samples for the determination of Polycyclic Aromatic Hydrocarbons (PAH) using High Resolution Gas Chromatography / Low Resolution Mass Spectrometry (HRGC / LRMS).

SAMPLE RECEIPT AND STORAGE

The samples were received on the 27th of June 2014. The sample temperature upon receipt ranged from 4.9°C to 6.6°C, which was above the method recommendations (<4°C). This was judged not to significantly impact the data accuracy and the analysis was allowed to proceed. The samples were stored at -20°C in the dark prior to extraction and analysis.

REPORTING CONVENTIONS

The AXYS contract number assigned for internal tracking was 4540. The samples were assigned a unique laboratory identifier of the form LXXXXX-XX, where X is a numeral; all data reports reference this unique AXYS ID plus the client sample identifier. To assist in locating data, a table correlating the AXYS ID with the client sample number is included in this data package.

The following laboratory qualifiers appear in this data package:

J	=	indicates an estimated value where the concentration of the analyte is less than the LMCL but greater than the SDL
K	=	a peak was detected that did not meet all the criteria for identification as the target analyte; the reported value is the estimated maximum possible concentration of analyte present.
ND	=	identifies a compound that was not detected.

Results are reported to three significant figures, in units of nanograms per sample (ng/sample).

QA/QC NOTES

The samples and associated QC samples (a Lab Blank and a known sample called "Ongoing Precision and Recovery" (OPR)) were analyzed in a batch named WG47974. Samples and QC samples analyzed in an analysis batch were carried intact through the entire analytical process. The sample data were reviewed and evaluated in relation to the batch QC samples.

- Sample analyte concentrations are not blank corrected. Sample data should be evaluated with consideration of analyte levels in the Lab Blank (AXYS ID WG47974-101).

- By virtue of the isotope dilution/internal standard quantification procedures, data are recovery corrected for possible losses during extraction and clean up.
- All linearity, CAL/VER, OPR and labelled compound recovery specifications were met.

ANALYTICAL DISCUSSION

No analytical difficulty was encountered.

DATA PACKAGE

This data package is assigned a unique data package identification workgroup, DPWG48398. This ID is shown on the front page of the data package.

Included in the data package after the narrative is the following documentation:

- Sample Correlation Table
- Sample data report (organized by AXYS ID)
- Laboratory QC data reports
- Instrumental QC data reports (organized by analysis date)
- Accreditation Scope

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, except for the conditions detailed above. In addition, I certify, that to the best of my knowledge and belief, the data as reported are true and accurate. The following signature, on behalf of AXYS Analytical Services Ltd, authorizes the release of the data contained in this data package.

Signed: (Matthew) Ziqing Ou, PhD

August 19, 2014
Date Signed

Environment Canada

CORRELATION TABLE

PAH ANALYSIS

Lab Name: AXYS Analytical Services Ltd.	Project Manager: Jenny Pape
Project Name: Project 349 Oil Sands Passive SPMD	Contract No: 4540
	AXYS Method: MLA-021
Data Package Identification: DPWG48398	Program: SPMD Samples
Client Sample No.	Lab Sample ID
LAB BLANK	WG47974-101
OPR	WG47974-102
2013PN620044	L21633-30
2013PN620045	L21633-31
2013PN620117	L21634-34
2013PN620118	L21634-35
2013PN620119	L21634-36
2013PN620120	L21634-37
2013PN620121	L21634-38
2013PN620122	L21634-39
2013PN620124	L21634-40
2013PN620125	L21634-41
2013PN620127	L21634-42
2013PN620128	L21634-43
2013PN620130	L21634-44
2013PN620131	L21634-45
2013PN620209	L21636-5
2013PN620210	L21636-6
2013PN620211	L21636-7
2013PN620212	L21636-8

Appendix C. Case Narratives

Analytical Batch	Data Package	Project	Analytical Issue	Discussion
WG48050	DPWG48470	PN62	none	
WG48052	DPWG48585	PN62	none	
WG48099	DPWG48458	PN62	**L21636-18** lost ~200 uL out of 1000 uL of first rinse transferring onto silica column **L21635-32** lost ~10-15% of volume transferring 2nd rinse from C-tube to GC/MS vial	Since the sample loss was after labeled surrogates had been added there is no effect on the accuracy of the reported analyte concentrations. The effect on surrogate recoveries will also be minimal as the loss was a portion of the rinse of the container (after the sample had been transferred) and not a portion of the sample extract.
WG48108	DPWG48425	PN62	**L21633-7, -8 and -9.** Samples with broken lids and leakage of hexane from first dialysis. Estimated losses by weight 7, 7 and 10% of the total of the first dialysate repectively).	These losses will slightly affect accuracy of analyte concentrations. Assuming 80% of analytes are in the first dialysate this will result in a low bias in the PAH concentrations of these 3 samples estimated to be much less than 10%
WG48123	DPWG48459	PN62	**L21633-22** Strip 3 of 3, missed adding hexane for 2nd dialysis. 2nd dialysis done the next day. **L21636-23** lost ~200uL out of 900 uL from GCMS vial while preparing sample for running.	**L21633-22.** No expected effect. **L21636-23.** Since the loss was after labeled surrogates had been added there is no effect on the accuracy of the reported analyte concentrations. However reported surrogate recoveries will appear low by about 20%.

Analytical Batch	Data Package	Project	Analytical Issue	Discussion
WG48226	DPWG48622	PN52	**L21658-11**. About 20% of sample extract spilled before splitting and cleanup.	Since labeled surrogates had been added to the extract before the spill occurred there will be no impact on quantification of analytes in the sample but the surrogate recoveries will be low by about the same amount as the spill (estimated at 20%)
WG48160	DPWG48633	PN53	**L21656-41**. The second dialysis of one of the 3 SPMD strips of this sample was carried out at 4°C rather than 67 °F. An extra hour of dialysis at 67 °F was then conducted to compensate for the slower dialysis at the cooler temperature. **L21656-8** received a double spike of surrogate before cleanup. **L21656-16** was not spiked with surrogate until after the extract had been concentrated by rotary evaporation (protocol is to add surrogates before concentration step).	**L21656-41**. No significant effect is expected as it was the second dialysis of only one third of the sample and extra dialysis time was added to ensure complete dialysis. **L21656-8.** The analyst had noted the spiking error and it was taken into account in the calculations so the reported results are accurate. **L21656-16. Since t**he surrogates were added after rotary evaporation rather than before any loss of volatile components during this step would not be adequately compensated for. There might therefore be a low bias in the concentrations of the most volatile PAH in this sample (e.g. naphthalenes).
WG48370	DPWG48623	PN54	**L21669-16**. The lab blank was inadvertently combined with this sample immediately after dialysis. Workup of the	There should be no significant effect from this error as it occurred so early in the processing of the sample. If the

Analytical Batch	Data Package	Project	Analytical Issue	Discussion
WG48388	DPWG48657	PN54	The first dialysis was carried out for 44 hours rather than the specified 18 hours **L21669-20, -24, -28, -41, -43**. These 5 samples were analyzed from a higher final volume than normal (550 to 600 µL rather than the standard 500 uL) because they were difficult to concentrate any further, likely due to material extracted from the SPMD tubes during the extended dialysis.	The potential consequence of longer dialysis times is that the SPMD tubes can start to dissolve or release plastic materials that interfere with the analysis. Chromatographic interference from this material was not observed in any of the samples in this batch. **L21669-20, -24, -28, -41, -43.** The higher final volumes for these samples will affect detection limits by the same percentage (10 to 18% higher).
WG48460	DPWG48677	PN54	**L21669-48**. Glassware breakage (~85%) of sample was lost during the extract drying step. Therefore no ½ split performed and an archived portion is not available. **L21669-36**, lost ~500 uL out of 1000 uL of the 1st pentane rinse while transferring onto silica cleanup column.	**L21669-48**. Since the sample loss was after labeled surrogates had been added there is no effect on the accuracy of the reported analyte concentrations. However labeled surrogate recoveries will appear low. **L21669-36.** Since the sample loss was after labeled surrogates had been added there is no effect on the accuracy of the reported analyte concentrations. The effect on surrogate recoveries will also be minimal as the loss was a portion of a rinse of the flask (after the sample had been transferred) rather than half of the sample extract.

Appendix D. Partitioning Coefficients – LogKow Values Input to USGS Estimator

Octanol:water (LogKow) partitioning coefficients for PACs vary widely in the literature and originate from laboratory testing and mathematical models. Coefficients used in the USGS Estimator were selected based on experimental research (as recommended by Mackay 2006, Sangster 1989) and, in the absence of these, calculated using the USEPA KOWWIN calculator (http://www.epa.gov/opptintr/exposure/pubs/episuite.htm). The calculator models partitioning based on the molecular weight of the compounds. When compared to experimental values, the calculated values were typically within 0.4 units and deemed to be similar (Ahmed and Lanez 2009, Lu et al. 2008, Gombar and Enslein 1996).

Analyte	Log Kow	Analyte	Log Kow	Analyte	Log Kow
Naphthalene	**3.35**	C1-Biphenyls	**4.30**	1-Methylphenanthrene	**5.08**
Acenaphthylene**	**3.94**	C2-Biphenyls	**4.85**	3,6-Dimethylphenanthrene	**5.44**
Acenaphthene	**3.92**	C2-Naphthalenes	**4.37**	2,6-Dimethylphenanthrene	**5.44**
2-Methylfluorene	**4.56**	1,2-Dimethylnaphthalene*	**4.31**	1,7-Dimethylphenanthrene	**5.44**
C2 Phenanthrenes/Anthracenes	**5.44**	2,6-Dimethylnaphthalene	**4.31**	1,8-Dimethylphenanthrene	**5.44**
Fluorene	**4.18**	C3-Naphthalenes	**4.73**	C3-Phenanthrenes/Anthracenes	**5.99**
Phenanthrene	**4.52**	2,3,6-Trimethylnaphthalene*	**4.73**	1,2,6-Trimethylphenanthrene	**5.99**
Anthracene	**4.45**	2,3,5-Trimethylnaphthalene	**4.73**	Retene	**6.35**
C1 Phenanthrenes/Anthracenes	**4.89**	C4-Naphthalenes	**5.36**	C4-Phenanthrenes/Anthracenes	**6.53**
Fluoranthene	**5.20**	C1-Acenaphthenes	**4.57**	C1-Fluoranthenes/Pyrenes	**5.48**
Pyrene	**5.00**	C1-Fluorenes	**4.97**	3-Methylfluoranthene/Benzo[a]fluorene	**5.48**
Benz[a]anthracene	**5.91**	1,7-Dimethylfluorene	**5.11**	C2-Fluoranthenes/Pyrenes	**6.03**
Chrysene	**5.86**	C2-Fluorenes	**5.11**	C3-Fluoranthenes/Pyrenes	**6.57**
Benzo[b]fluoranthene	**5.78**	C3-Fluorenes	**5.24**	C4-Fluoranthenes/Pyrenes	**7.12**
Benzo[j,k]fluoranthenes**	**6.40**	Dibenzothiophene	**4.38**	C1-Benzo[a]anthracenes/Chrysenes	**6.07**
Benzo[e]pyrene	**6.44**	C1-Dibenzothiophenes	**4.71**	5/6-Methylchrysene	**6.07**
Benzo[a]pyrene	**6.35**	2/3-Methyldibenzothiophenes	**4.71**	1-Methylchrysene	**6.07**
Perylene	**6.25**	C2-Dibenzothiophenes	**5.26**	C2-Benzo[a]anthracenes/Chrysenes	**7.20**
Dibenz[a,h]anthracene	**6.75**	2,4-Dimethyldibenzothiophene	**5.26**	5,9-Dimethylchrysene	**6.62**
Indeno[1,2,3-cd]pyrene**	**6.72**	C3-Dibenzothiophenes	**5.81**	C3-Benzo[a]anthracenes/Chrysenes	**7.16**
Benzo[g,h,i]perylene	**6.90**	C4-Dibenzothiophenes	**6.35**	C4-Benzo[a]anthracenes/Chrysenes	**7.70**
2-Methylnaphthalene	**4.00**	3-Methylphenanthrene*	**5.15**	C1-Benzofluoranthenes/Benzopyrenes	**6.66**
1-Methylnaphthalene	**3.87**	2-Methylphenanthrene*	**5.24**	7-Methylbenzo[a]pyrene	**6.66**
C1-Naphthalenes	**3.86**	2-Methylanthracene	**5.00**	C2-Benzofluoranthenes/ Benzopyrenes	**7.20**
Biphenyl	**3.98**	9/4-Methylphenanthrene	**4.89**	1,4,6,7-Tetramethylnaphthalene	**5.36**

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