

Inland Empire Paper Company

**NPDES Permit WA-0000082-5
Special Condition S8.A.7**

Quality Assurance Project Plan (QAPP)

**Source Control and Effluent Characterization
in Support of the PCB Pollutant Minimization
Plan (PCB PMP)**

**December 18, 2024
Version 2.0**

TABLE OF CONTENTS

TABLE OF CONTENTS	2
REVISION HISTORY	3
PURPOSE	4
ORGANIZATION	6
QUALITY CONTROL OBJECTIVES	7
SAMPLING PROTOCOLS.....	8
SAMPLE SCHEDULE	8
SAMPLING QUALITY ASSURANCE	8
SAMPLE TYPE	8
SAMPLING LOCATION	9
SAMPLING PROCEDURE	9
LABORATORY PROTOCOLS AND QUALITY CONTROL	11
SAMPLE RECEIVING AND HOLDING.....	11
QUALITY CONTROL RESPONSIBILITY.....	11
TERMINOLOGY	11
QUALITY CONTROL CRITERIA	12
DATA REPORTING AND QUALIFIERS	13
DATA INTERPRETATION AND MANAGEMENT	15
DOCUMENTATION AND RECORDKEEPING	15
INTERPRETIVE METHOD	15
FLAG QUALIFICATION	16
BLANK CENSORING	16
PRECISION ANALYSIS	17
ACCURACY ANALYSIS.....	20
SUMMATION	20
REPORTING	22
 APPENDIX A	 Sampling and Analysis Plan (SAP) Worksheets
APPENDIX B	Eurofins Sacramento Chain of Custody Form - Template

REVISION HISTORY

<u>Date</u>	<u>Version</u>	<u>Summary of Changes</u>
8/1/2023	1.0	Original submission of QAPP to Ecology.
12/18/2024	2.0	Changed laboratory to Eurofins Sacramento. Clearer instructions on sample handling and quality control samples. Clearer instruction on data verification and reporting.

PURPOSE

National Pollutant Discharge and Elimination System (NPDES) Permit number WA-0000082-5 Special Condition S8.A requires the permittee, Inland Empire Paper Company (IEP), to submit a Polychlorinated Biphenyl (PCB) Pollutant Minimization Plan (PMP) to characterize PCBs in IEP's effluent. Special Condition S8.A.7 further clarifies that the data collected and analyzed for the PCB PMP must be performed within the terms of a Quality Assurance Project Plan (QAPP) and the PCB analysis method must be performed using EPA Method 1668C. The specific permit language is as follows:

7. Quality Assurance/Quality Control (QA/QC) Plan for PCB source control and effluent characterization. The QA/QC Plan must include a minimum testing frequency of once per quarter for routine monitoring of PCBs in the final effluent (Outfall 001) for effluent characterization using EPA method 1668. Prepare the QA/QC Plan in accordance with the guidelines provided in Guidelines for Preparing Quality Assurance Project Plans for Environmental Studies, Ecology publication 04-03-030.¹

As stated in Special Condition S8.A.7 above, the data collected under the terms of this QAPP may be used only for "PCB source control and effluent characterization" and is not intended for application of the numeric PCB water quality criteria. Ecology had codified this policy, with approval from the Environmental Protection Agency (EPA), in the Washington Administrative Code (WAC):

(3) Procedures for applying water quality criteria. In applying the appropriate water quality criteria for a water body, the department will use the following procedure:

...

(h) The analytical testing methods for these numeric criteria must be in accordance with the "Guidelines Establishing Test Procedures for the Analysis of Pollutants" (40 C.F.R. Part 136) or superseding methods published. The department may also approve other methods following consultation with adjacent states and with the approval of the USEPA.²

Ecology has not only acknowledged that EPA Method 1668C has not been approved by EPA for compliance purposes, but also that the test method is unreliable. Ecology's Permit Writer's Manual states:

¹ NPDES Permit No. WA00000825. State of Washington, Department of Ecology, Eastern Regional Office. Effective date August 1, 2022. Page 28.

² Natural Conditions and Other Water Quality Criteria and Applications. WAC 173-201A-260 (2011). <https://app.leg.wa.gov/wac/default.aspx?cite=173-201A-260>

Method 1668C is not currently approved by EPA for effluent limit compliance under 40 CFR Part 136. And, Ecology is not proposing to seek EPA approval of this method under 40 CFR 16.5 as there are known problems in regards to the repeatability and accuracy of the method in addition to the expense of the analysis.³

In summary, the two controlling regulatory authorities, Ecology and EPA, begin from a premise lacking confidence in the underlying analytical method, yet expect IEP, as the permittee, to extract reliable and quantifiable data from it.

To fulfill this contradictory mandate, this QAPP relies on best scientific practices of data collection and analysis to identify data that is truly quantifiable, while minimizing consideration of unreliable data.

³ Department of Ecology. Water Quality Program Permit Writer's Manual (2018). Pub. No 92-109. Page 226.

ORGANIZATION

The QAPP is written and implemented primarily by IEP's Technical Department, in collaboration with IEP's Environmental Manager. Members of IEP's cross-functional team include:

Benjamin Carleton – Technical Superintendent
David Demers – Process Technician
Doug Krapas – Environmental Manager

Inland Empire Paper Company
3320 N. Argonne Road
Spokane, WA 99212
(509) 924-1911

Professional consultation and review of the QAPP is provided by Exponent. Professional laboratory services are provided by Eurofins Sacramento:

Exponent
15375 SE 30th Place
Suite 250
Bellevue, WA 98007
(425) 519-8700

Jill Kellmann
Eurofins Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
(916) 374-4402

QUALITY CONTROL OBJECTIVES

Special Condition S8.A.7 defines the objective of the QAPP to be “for PCB source control and effluent characterization.” The permit further requires the use of EPA Method 1668C because, among all PCB methods, it has the highest degree of sensitivity, with published values in the range of 7-77 parts per quadrillion (picograms per liter, pg/L) per congener in clean water.⁴ However, the high sensitivity comes with a greater degree of risk of background interference or contamination. Method 1668C has failed to be promulgated in the Code of Federal Regulations (CFR) at 40 CFR Part 136 as a compliance method under the Clean Water Act because of these concerns.⁵

The primary data quality objective of this QAPP is to obtain data of the highest possible sensitivity while also being truly quantifiable and scientifically defensible. This objective will be met with the following data quality indicators⁶:

- **Sensitivity**. The capability of a method or instrument to discriminate between small differences in analyte concentration and the qualitative description of an analytical method’s detection limit.
- **Accuracy**. The degree of agreement between an observed value and an accepted reference or “true” value.
- **Precision**. A measure of how closely values from replicate measurements of a sample agree with each other.
- **Representativeness**. The degree to which data accurately and precisely represent a characteristic of a population, sampling point, process condition or environmental condition.
- **Comparability**. The degree to which different methods or data can be compared and agree, or can be represented as similar.
- **Completeness**. A measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained.

⁴ U.S. Environmental Protection Agency. Method 1668C Chlorinated biphenyl Congeners in water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (April 2010). Pub. No EPA-820-R-10-005.

⁵ Department of Ecology. Water Quality Program Permit Writer’s Manual (2018). Pub. No 92-109. Page 226.

⁶ Definitions of data quality indicators derived from EPA Environmental Sampling and Analytical Methods Program glossary. <https://www.epa.gov/esam/glossary>.

SAMPLING PROTOCOLS

Sample Schedule

Special Condition S8.A.7 of the NPDES permit requires sampling to be conducted once at least once per quarter. IEP will sample once in each quarter according to the standard calendar definition:

- Q1 – January to March
- Q2 – April to June
- Q3 – July to September
- Q4 – October to December

Reporting of results to Ecology is to be conducted annually as part of the overall submittal package of IEP's annual update to the PCB PMP. See below in section *Reporting* for more detail.

Sampling Quality Assurance

Prior to each sampling event, the laboratory will provide the following testing kit to IEP:

- A shipment-ready cooler
- Two bottles for the field sample (FS)
- Two bottles for field duplicate (optional)
- Two bottles for the field blank (FB)
- Source water for the field blank
- Paper copy of chain-of-custody (COC)
- Custody seals

The laboratory will provide the sampling materials to minimize contamination and promote repeatability by standardizing the preparation process.

Additionally, two quality control samples are to be collected and shipped routinely:

- Field duplicate – at least once annually
- Field blank – every quarter

The purpose of the field duplicate is to estimate the variability of the individual results during the sampling and analytical procedures. Field duplicate will be analyzed at least once per year.

The purpose of the field blank (also called the transfer blank) is to verify that contamination did not occur during sample collection, preservation, shipment, or during the extraction and analysis at the laboratory.

Sample Type

All samples are collected as grab samples, not composite samples. Composite samples for PCB analysis introduce an unacceptable risk of cross-contamination that outweighs the marginal increase in representativeness. The biggest quality assurance risk of grab samples is the possibility of them being non-representative over a period of time. This is adequately compensated by the collection of a field duplicate and restricting sampling to times of typical operating conditions of the mill-site.

Documentation of prevailing mill conditions are kept in a field log for each sampling event and record, including the following information:

- Date and time of sample collection
- Personnel that performed the sample collection and the responsibility of each
- Paper grade production at the time of sample collection
- Pulp mill blend ratios to the blend chest and to the mix chest at the time of collection
- Approximate 1-hour effluent flow through the Parshall flume at the time of collection
- Approximate 1-hour sum of non-contact cooling water flows at the time of collection

The documentation of this information facilitates the collection of samples under conditions that are within normal mill operating parameters. The field log notebook is hard copy only and resides at the IEP Laboratory.

Sampling Location

Special Condition S8.A.7 of the NPDES permit requires “effluent characterization” at Outfall 001. This Outfall is the confluence of IEP’s non-contact cooling water (Outfall 004) with the treated process wastewater, and is representative of the final discharge to the Spokane River.

Sampling Procedure

The COC form provided by the lab will be filled out in advance with the sample identification, date, time, company, and sampler’s initials. The laboratory will be notified at least three business days in advance that IEP intends to collect a new set of samples to facilitate proper receipt upon arrival.

Contamination is a concern in both sampling and analysis for PCBs, therefore special care must be taken towards sample collection. Sampling involves two people using the Clean Hands/Dirty Hands procedure in order to avoid cross- contamination from the surrounding environment. This procedure is described in the *Interagency Field Manual for the Collection of Water-Quality Data* (USGS 2000) and U.S. Environmental Protection Agency Method 1669, *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (USEPA 1996).

- For the field samples and field blank, prior to sample collection, place each lab-provided glass bottle (pre-labeled) into two nested one-gallon plastic zipper bags. Two pairs of new nitrile gloves will be set aside per person in another new plastic bag and sealed.
- At the time of collection, the sealed bags containing the sample bottle and the gloves will be carried out to the collection site by the two designated samplers. The person who is designated Dirty Hands will open the sealed bag of gloves allowing the person designated as Clean Hands to don their gloves first before putting on their own gloves. Both people will put on two pairs of gloves handling the second pair only after the first pair is fully worn.
- Dirty Hands will then take the sample bottle and open the outer seal being careful to touch only the outer plastic bag.
- Clean Hands will then open the inner seal and remove the sample bottle from the inner plastic bag being careful not to touch the outer bag.

- The field blank will be collected prior to collection of field samples. During sampling, take the labeled field blank bottle and open it near where samples are collected. Fill the container with the lab-provided field blank water and close the bottle with clean gloves.
- For the field samples, Dirty Hands opens the access hatch on the Parshall flume so that Clean Hands can retrieve a grab sample with the glass bottle without contacting anything else.
- Once the field sample has been collected and the bottle is again capped, Clean Hands is responsible to seal the bottle inside the inner plastic bag avoiding contact again with the outer bag.
- Dirty Hands then seals the outer bag without touching the inside bag.
- The samples are brought back to IEP's lab and temporarily stored in the laboratory refrigerator while preparations are made for immediate shipment.
- Samples are to be shipped overnight priority to the laboratory in the same cooler provided, along with a copy of the COC. Samples must be shipped with ice packs to remain cool while in transit.
- Custody seals are to be placed over the cooler to ensure it is not tampered with during transportation.
- An electronic copy of the COC is transmitted to the laboratory Project Manager while the cooler is in transit.

LABORATORY PROTOCOLS AND QUALITY CONTROL

Sample Receiving and Holding

Upon arrival of a sampling set from IEP, the Eurofins Sacramento lab will receive and document the contents according to routine laboratory protocol. IEP will normally request the standard turnaround time (TAT), which is approximately 20 days for the final report. Any change to TAT will be arranged in advance with the lab and noted on the COC.

Maximum holding time for preserved samples is 365 days, however, IEP has arranged for samples to be held for only 30 days after receipt of the final report. This hold time allows IEP to request repeat analyses from the lab after reviewing the data.

Quality Control Responsibility

The laboratory is responsible for adhering to all quality control criteria as defined in this QAPP and in accordance with internal laboratory standards. This includes taking the corrective actions as defined in Appendix A if any criteria are not met.

If quality control criteria are not met, even after taking the specified corrective actions, the laboratory is to affix the appropriate qualifiers and submit all data results for samples and QC samples. It is IEP's responsibility to appropriately interpret the meaning and validity of the data, in accordance with the procedures outlined in the section *Data Interpretation and Management*.

Terminology

Reoccurring terms related to quality control samples and criteria are defined here for ease of reference.

Field duplicate (FD).

A second sample taken in the field at the same time as the original, with the same tools and protocols. Field duplicates detect precision in sampling and laboratory techniques. This is distinguished from a laboratory duplicate (LD), which splits a single field sample into two sub-samples. LDs are a measure of laboratory precision, but not field precision.

Method Blank (MB).

A sample of purified, analyte-free matrix (e.g. water) is taken through every step of the method. MBs detect contamination in the laboratory extraction and analytical procedures.

Isotope Dilution Analyte (IDA).

Stable, isotope-enriched analytes are added to the sample and act as an internal standard in mass spectrometry. The IDA can adjust the results for sample matrix effects.

Laboratory control sample (LCS).

A sample of purified, analyte-free matrix (e.g. water) is fortified with a known, verifiable quantity of analyte and then carried through the extraction and analytical methods for quantification. A

lab-fortified blank (LFB) or ongoing precision and recovery standard (OPR) are other references for an LCS. An LCS duplicate is abbreviated as LCSD and may be performed in addition to the LCS

Estimated Detection Limit (EDL).

A sample-specific estimate of the analyte concentration that would need to be present to generate a signal-to-noise ratio of at least 2.5. The EDL is similar in principle to a method detection limit (MDL), but unlike a MDL, the EDL cannot be determined in advance because it is sample-specific.

Limit of Quantitation (LOQ).

The minimum concentration that can be reported as quantifiable. Defined to be equal to the lowest calibration standard for the analyte, or, alternatively, the concentration that would need to be present to generate a signal-to-noise ratio of 10. Also known as the quantification limit (QL) or minimum reporting limit (MRL).

Percent Recovery (PR).

A measurable quality control criteria in which a known quantity of an analyte in a sample matrix is measured, or recovered, by analysis. Measures accuracy and is appropriate for IDA and LCA.

$$PR = \frac{\text{Measured concentration}}{\text{Known concentration}} \times 100\%$$

The lower bound for acceptance of the PR is the lower control limit (LCL), and the upper bound for acceptance of the PR is the upper control limit (UCL).

Relative Percent Difference (RPD).

A measurable quality control criteria in which the difference between two duplicates is divided by the average of the two results. Measures precision and is appropriate for FD.

$$RPD = \frac{|Original - Duplicate|}{\left(\frac{Original + Duplicate}{2}\right)} \times 100\%$$

Quality Control Criteria

Appendix A contains the detailed information for the system analysis plan (SAP) that the laboratory will follow. A list of the attachments include:

- Worksheet 1 – reference limits for LOQs and EDLs, per congener, for aqueous samples. This worksheet also includes the co-elution list. Note that EDLs are approximate only, and are determined individually for each sample.
- Worksheet 2 – analytical standard operating procedure requirements.
- Worksheet 3 – analytical standard operating procedure references.
- Worksheet 4 – analytical instrument calibration table. Includes instrument specific acceptance criteria and corrective actions.
- Worksheet 5 – analytical instrument equipment maintenance, testing, and inspection.

- Worksheet 6 – laboratory QC samples table. Includes the frequency, acceptance criteria, and corrective actions.
- Worksheet 7 – method-specific LCLs/UCLs for LCS and IDA analyses.

The list of quality control samples from Worksheet 6, including frequency, criteria, and corrective action, is summarized below:

<u>QC Sample</u>	<u>Frequency</u>	<u>Criteria</u>	<u>Corrective Action</u>
Method blank	Once per preparation batch	No target analytes (i.e. individual congeners) greater than the individual LOQ.	Verify instrument is clean and reanalyze
Isotope dilution analyte	Every sample	Recovery limits defined by Method 1668C. 5-145%, 10-145% for samples; 15-145%, 40-145% for LCS.	Reprep and reanalyze samples with failed criteria
Laboratory control sample	Once per preparation batch	Recovery limits defined by Method 1668C. 60-135%.	Reanalyze LCS once, report results from LCS and LCSD. If failed again, reprep and reanalyze all samples if sufficient material remains. Otherwise, report results as is.
Field duplicate	As requested by IEP, minimum once per year.	See section <i>Precision Analysis</i> . Responsibility of IEP.	See section <i>Precision Analysis</i> . Responsibility of IEP.

Data Reporting and Qualifiers

Eurofins Sacramento will deliver to IEP a Level II electronic data deliverable (EDD) in PDF format and spreadsheet format compatible with Microsoft Excel.

Analytical results are reported per each individual congener (except co-elutions). Summations of homolog groups or total PCBs may be included in the report for illustrative, but unofficial, purposes. Individual values may be flagged and/or qualified according to the following table.

<u>Qualifier Flag</u>	<u>Definition/Description</u>
J	The reported result is an estimate. The value is greater than the EDL but less than the LOQ.
U	The analyte was not detected in the sample at a concentration greater than the EDL.
E	Analyte exceeds the instrument calibration range.
D	Dilution data. Result was obtained from the analysis of a dilution
B	Analyte found in both the sample and the associated method blank
q	Estimated maximum possible concentration. Indicates that a peak is detected but did not meet all the method required identification criteria.

DATA INTERPRETATION AND MANAGEMENT

Documentation and Recordkeeping

IEP will receive and file only electronic versions of data generated under this QAPP. The files will be saved on a server located at IEP's physical premise. All of IEP's servers are backed up in full at least once per month and stored offsite in the event of data loss or security breach.

The PDF versions of the data reports are non-editable and contain the raw data in its non-interpreted form. The electronic data deliverables (EDDs) will be compatible with Microsoft Excel and are intended to facilitate data interpretation, including summations, as described in the rest of the section. The raw data in the EDDs will not be intentionally overridden, but because of the editable nature of the software, there is a chance of discovering a discrepancy between the EDD and PDF report. In that case, IEP will contact the laboratory Project Manager to confirm the accuracy of the PDF data, including resending another copy of the report if necessary. Once confirmed, the EDD will need to be updated and recalculated.

Data will be retained on the server for at least three years in accordance with NPDES Permit WA-000082-5 Special Condition S3.C:⁷

Interpretive Method

EPA Method 1668C utilizes GC-MS to independently determine the concentration of individual congeners, with some exceptions for co-elutions. Therefore, the proper interpretive approach is to qualify the data result of each individual congener as if it were an individual analyte. Summary values, especially summations for common groups like homologs or total PCBs, are the last step in the interpretation process.

To avoid confusion, congeners that co-elute will be reported and handled as a single result, as if it were only a single congener. The number and identity of each congener in the co-elution will be listed in the identification name. This reporting method for co-elution guards against duplication and double-counting in the summation step.

Each sample result, which contains information for each individual congener, must be handled identically, regardless of whether the matrix was a field sample or QC sample. The order of operations for data interpretation is as follows:

1. Flag qualification
2. Blank censoring
3. Precision analysis
4. Accuracy analysis
5. Summation

⁷ NPDES Permit No. WA00000825. State of Washington, Department of Ecology, Eastern Regional Office. Effective date August 1, 2022. Page 19.

Flag Qualification

Definitions of flags is found in the previous section

Data Reporting and Qualifiers. In this section, it is defined whether, and how, flag qualifiers will be interpreted under this QAPP.

<u>Qualifier Flag</u>	<u>Handling</u>
J	Indicates an actual detection of the analyte, but only an estimation. Values are subject to precision analysis (see subsequent section <i>Precision Analysis</i>)
U	Considered to be non-detect. Convert any reported numeric values to zero.
E	The reported value exceeds the instrument calibration range and should be used as an estimated value.
D	Result was reported from a diluted sample. No action needed and the result should be used as reported by the lab.
B	Method blank detection is handled with blank censoring (see subsequent section <i>Blank Censoring</i>).
q	Indicates that some, but not all, criteria for a positive identification were met. Convert any reported numeric values to zero. ⁸

Conversion of 'U' and 'q' flags to a numerical value of zero will occur prior to the subsequent steps in the data interpretation process.

Blank Censoring

NPDES Special Condition S8.A.7 specifies that reporting of data under the QAPP must include blank censoring at tier levels of zero, five, and ten. Blank censoring is defined as converting to a numeric value of zero any individual congener concentration in the field sample that is less than the censor tier in the associated blank. Because this QAPP will always have a field blank and a method blank, the censoring will be against the maximum value of both blanks for each individual congener. The following example demonstrates the method:

Blank Censor Example:	Congener X, field sample (FS) = 234 pg/L
	Congener X, field blank (FB) = 10 pg/L
	Congener X, method blank (MB) = 25 pg/L

⁸Environmental Protection Agency. OSRTI. National Functional Guidelines for High Resolution Superfund Methods Data Review (2020). Pages 40-42. <http://www.epa.gov/clp/contract-laboratory-program-national-functional-guidelines-data-review>.

0x censoring:	(FS < 0*FB) OR (FS < 0*MB) (234 < 0) OR (234 < 0) (FALSE) OR (FALSE) FALSE Report Congener X = 234 pg/L
5x censoring:	(FS < 5*FB) OR (FS < 5*MB) (234 < 50) OR (234 < 125) (FALSE) OR (FALSE) FALSE Report Congener X = 234 pg/L
10x censoring:	(FS < 10*FB) OR (FS < 10*MB) (234 < 100) OR (234 < 250) (FALSE) OR (TRUE) TRUE Report Congener X = 0 pg/L

From the example above, Congener X retained its value at tier levels of zero and five, but was converted to a concentration of zero at tier level ten. Blank censoring at a tier level of zero is a trivial case and will always result in no adjustment to the concentrations, and does not need to be manually calculated.

Blank censoring at the three required tier levels of zero, five, and ten results in three subsets of data generated for each individual sample. The next two data qualification steps, precision analysis and summation, are independently conducted on each of these sub-sets.

Precision Analysis

At this stage of the analysis, values less than the EDL have been screened out as non-detect from the *Flag Qualification* step. Therefore, any remaining 'J'-flagged data is a true detection that must be reported, but because it is less than the LOQ, cannot be confidently quantified. These competing yet equally important data quality objectives – sensitivity and precision – are difficult to reconcile for 'J'-flagged data. IEP's approach to this challenge is to assign numeric values to congeners so that summation is possible, but sufficiently qualified to provide the full context of quality assurance. The process differs slightly depending on whether a duplicate sample was analyzed.

Single Sample, No Duplicate

For individual samples with no duplicate, the data will be compiled and reported as a VALUE and a QUALIFIED VALUE. For every congener, the VALUE is the analytical value reported by the lab after screening for *Flag Qualification* and *Blank Censoring*. For every 'J'-flagged congener, the QUALIFIED VALUE is also the post-screening analytical value, but for congeners without a 'J' flag, the QUALIFIED VALUE is zero. See the table and example below for illustration.

<u>Result Combination (per congener)</u>	<u>Measurement Quality Objective (MQO)</u>	<u>Report Action if MQO is PASSED</u>	<u>Report Action if MQO is FAILED</u>
Single sample result greater than LOQ	No quality issues with the result; MQO not applicable	VALUE = analytical result QUALIFIED VALUE = 0	N/A
Single result less than LOQ	'J' flagged, by definition, estimate only; MQO not applicable	N/A	VALUE = analytical result QUALIFIED VALUE = analytical result

Single Sample, No Duplicate

Precision Analysis Example:

Congener A = 31 pg/L (J)
 Congener B = 75 pg/L (J)
 Congener C = 398 pg/L

Precision Test: No duplicate; MQO not applicable

	VALUE (pg/L)	QUALIFIED VALUE (pg/L)
Congener A	31	31
Congener B	75	75
Congener C	398	0

Duplicate Samples

For samples that are analyzed in duplicate, the data will be compiled and reported as MEAN, MIN, and MAX. For every congener, the MEAN is the arithmetic mean of the analytical result of each duplicate pair after screening for *Flag Qualification* and *Blank Censoring*.

The values assigned for MIN and MAX depend on whether any values are 'J'-flagged (i.e. less than LOQ) and whether the Measurement Quality Objective (MQO) passes or fails the RPD criteria. For example, if two duplicate results are both less than LOQ, but the RPD criteria is satisfied, then there is confidence that the true value lies between the min and max value of the duplicate pair. Conversely, if the duplicate results are both less than the LOQ, but the RPD criteria is not satisfied, then the best that can be known is that a detection lies somewhere between the EDL (i.e. point of non-detect) and the LOQ (i.e. point of quantification).

The table below provides instruction on how to handle each combination, and the examples below illustrate the procedure.

<u>Result Combination (per congener)*</u>	<u>Measurement Quality Objective (MQO)</u>	<u>Report Action if MQO is PASSED</u>	<u>Report Action if MQO is FAILED</u>
Two duplicates greater than the LOQ	RPD <= 30%	MIN = arithmetic mean MAX = arithmetic mean	MIN = min value MAX = max value
One duplicate greater than LOQ, and one duplicate less than LOQ	RPD <=30%	MIN = arithmetic mean MAX = arithmetic mean	MIN = min value MAX = max value
Two duplicates less than LOQ	RPD <= 30%	MIN = min value MAX = max value	MIN = EDL MAX = LOQ
One duplicate equal to zero, and one duplicate greater than zero	RPD <=30%	N/A; RPD = 200% by definition	MIN = 0 MAX = max value
Two duplicates equal to zero	No quality issues with the result; MQO not applicable	MIN = 0 MAX = 0	N/A

*NOTE: In all cases, the MEAN value is assigned the arithmetic mean of the duplicate pair

Duplicate Samples

Precision Analysis Example:

Congener A dup. 1 = 31 pg/L (J)
 Congener A dup. 2 = 188 pg/L (J)
 EDL = 2.5 pg/L
 LOQ = 200 pg/L

Congener B dup. 1 = 75 pg/L (J)
 Congener B dup. 2 = 90 pg/L (J)
 EDL = 1.5 pg/L
 LOQ = 200 pg/L

Congener C dup.1 = 398 pg/L
 Congener C dup. 2 = 175 pg/L (J)
 EDL = 5.0 pg/L
 LOQ = 200 pg/L

Precision Test: Congener A RPD <= 30%
 143% <= 30%
 FALSE

Congener B RPD <= 30%

18% <= 30%

TRUE

Congener C RPD <= 30%

78% <= 30%

FALSE

	MEAN (pg/L)	MIN (pg/L)	MAX (pg/L)
Congener A	109.5	2.5	200
Congener B	82.5	75	90
Congener C	286.5	175	398

Accuracy Analysis

The accuracy of the sample results, including corrective action, is handled primarily by the laboratory according to the laboratory SAP (see Appendix A). If the initial LCS fails, the lab is responsible for reanalyzing the LCS and re-extracting the samples. However, one of two conditions may occur:

- Initial LCS fails to meet all QC criteria, and re-analysis of LCS also fails to meet all QC criteria.
- Initial LCS fails to meet all QC criteria, but there is insufficient volume to re-extract samples and must be reported as-is (regardless of whether re-analysis LCS succeeds or fails).

If one of these conditions prevails, there is no confidence in the accuracy of the sample data, and the data cannot be used to establish a quantifiable concentration of PCBs in the effluent. In this case, no numeric value can be assigned to any congener and no data will be reported.

In order to justify reporting a null result, the specific MQO criteria must be highly conservative and protective of the dual DQIs accuracy and sensitivity. One of two conditions must be met:

1. Any single congener in the LCS has a percent recovery of less than 10% (method-specific acceptance range is 60-135%; see, e.g., *Quality Control Criteria under Laboratory Protocols and Quality Control*)
2. If 50% of the congeners in the LCS are outside of the method specific acceptance range of 60-135%

Summation

Provided all the previous data qualification steps are conducted systematically, and in the order described, summation is a straightforward process. By default, IEP will sum each homolog group and total PCBs.

For single samples with no duplicate, the VALUE and QUALIFIED VALUE are summed separately and reported independently. For duplicate samples, the MEAN, MIN, and MAX are summed separately and reported independently. The process is repeated for each tier of blank censoring.

Summation Examples:**Single sample, no duplicate**

	VALUE (pg/L)	QUALIFIED VALUE (pg/L)
Congener A	31	31
Congener B	75	75
Congener C	398	0

SUM (A->C)	504	106

Duplicate samples

	MEAN (pg/L)	MIN (pg/L)	MAX (pg/L)
Congener A	109.5	2.5	200
Congener B	82.5	75	90
Congener C	286.5	175	398

SUM (A->C)	478.5	252.5	688

As discussed in the section *Precision Analysis*, the intent behind summing QUALIFIED VALUE, MIN, and MAX in addition to VALUE and MEAN is to provide full quality assurance context while still being able to assign and compile numeric values. The relative importance and implications of these qualifiers is outside the scope of this QAPP and is intended to be discussed within the Annual Status Update (see *Reporting*).

REPORTING

IEP will report data collected under this QAPP annually in conjunction with the Annual Status Update to the PCB PMP. The report itself will contain summary information on homologs and total PCBs with blank censor levels of zero, five, and ten as required by Special Condition S8.A.7 and the qualification procedures outlined in this QAPP, especially those discussed in the sections *Precision Analysis* and *Summation*. Elsewhere in the report, IEP will preferentially utilize the sub-set of data that is blank censored at a tier level of ten to be consistent with the Department of Ecology's policy as stated in the Permit Writer's Manual:

Using 10x censoring for summation of the 209 PCB congeners removes false positives that are not significantly above (e.g. less than 2 standard deviations from the mean) the blank level. The value of 10x equates to 95% confidence level that the congener is present in the sample and is also quantifiable.⁹

A copy of the modified data consistent with the steps described in the previous section *Data Interpretation and Management* will be included as an appendix to the PCB PMP. Because of the large amount of data and file size, the PDF reports and unedited spreadsheet files will not ordinarily be reported with the PCB PMP, but will remain available for review upon request or for auditing purposes in accordance with the policy set forth in the section *Documentation and Recordkeeping*.

Official reporting will be through the Water Quality Permitting Portal (WQWebPortal), administered by Ecology, and accessed through the Secure Access Washington (SAW) web application. A public record of this QAPP may be accessed and viewed via the Permitting and Reporting Information System (PARIS), also administered by Ecology.

⁹ Department of Ecology. Water Quality Program Permit Writer's Manual (2018). Pub. No 92-109. Page 225.

APPENDIX A

Sampling and Analysis Plan (SAP) Worksheets

APPENDIX B

Eurofins Sacramento Chain of Custody Form – Template