

Quality Assurance Project Plan

Proposal to Pilot the Inadvertent PCB (iPCB) Pigment Resource to Develop No or Ultra-Low iPCB Inks for Printing

by:

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1.3 Distribution List

- 1. Doug Krapas, Environmental Manager of Inland Empire Paper and Chair of the TSCA Workgroup of the Spokane River Regional Toxics Task Force: <u>dougkrapas@iepco.co</u>
- 2. Benjamin Floyd, Facilitator of the Spokane River Regional Toxics Task Force: <u>ben@whitebluffsconsulting.com</u>

2.0 Abstract

Polychlorinated biphenyls (PCBs) are a class of chemicals with 209 congeners known to contaminate the Spokane River. Some PCBs within the river come from PCBs that are inadvertently generated and that are present in consumer products. Inadvertently generated PCBs (iPCBs) are byproducts of manufacturing other chemicals, including pigments. They differ from "legacy PCBs" that were commercially generated for use in multiple applications including use in electrical transformers, plasticizers, carbonless paper and many other applications. In the United States, PCBs were commercially manufactured from 1929 until production was banned in 1979 by the Toxic Substances Control Act (TSCA). However, EPA's regulations implementing TSCA for PCBs allow some inadvertent generation of PCBs to occur in excluded manufacturing processes, as defined in title 40 of the Code of Federal Regulations (CFR) section 761.3.

The Washington State Department of Ecology (Ecology), tested a selection of printing inks and found detectable levels of iPCBs. At the same time, in Spokane, WA, wastewater effluent from the recycling of printed paper exceeds required wastewater PCB limits. The allowable levels of PCBs in pigments (up to 50 ppm with an average of 25 ppm) creates challenges for paper manufacturers and recyclers who are held to wastewater effluent limits for iPCBs in the parts per quadrillion range.

This Quality Assurance Project Plan (QAPP) describes the planned testing procedures for a pilot project that is designed to address the problem of inadvertent PCBs in printed paper by going "upstream" to address their source. A proposal was developed and approved by the Spokane River Regional Toxics Task Force (SRRTTF) in the first quarter of 2023 to engage pigment and printing experts and to pilot a collaboration between an ink manufacturer, two printing facilities, and ChemFORWARD (project management). In this pilot, process inks currently used for newsprint by two printers within the Spokane "recycle shed" will be tested for iPCBs. One of the printers has agreed to trial a reformulated ink with a focus on yellow since the yellow ink is anticipated to have the highest levels of iPCBs. This printer agreed to trial the replacement of their existing yellow ink (presumed to contain inadvertently generated PCBs) with a reformulated ink that contains no, or ultra-low, levels of iPCBs because it was manufactured without the use of chlorinated reagents and/or auxiliaries. The project will leverage information in the ChemFORWARD Pigment Resource, a database that was developed with funding from the Spokane Regional Toxics Task Force (SRRTTF), to identify pigment alternatives that are NOT likely to contain iPCBs. The new ink formulation will be tested during development for iPCBs to ensure that it does indeed contain lower iPCB concentrations. The incumbent and alternative inks will also be evaluated for printing performance characteristics and for their cost structure within the printer's operational structure.

This QAPP has been prepared for approval by the SRRTTF. The SRRTTF will be dissolved as of 30 June 2023 and at that time, the remaining funds at SRRTTF will be transferred to Ecology. It is anticipated that the original pilot proposal that was approved by the SRRTTF and this QAPP will be submitted to Ecology after the 30 June 2023 deadline. There was not enough time to prepare

the QAPP and to complete the project work and PCB testing prior to the dissolution of the SRRTTF. Even though the SRRTTF approved funding for the proposed pilot project, Ecology is not obligated to fund the project. It is anticipated that the QAPP will be reviewed by Ecology when it is submitted with the proposal after 30 June 2023 and at that time, edits may be necessary.

3.0 Background

3.1 Introduction and problem statement

PCBs are a known contaminant in the Spokane River (SRRTTF, 2023). Studies have shown that some PCBs within the river potentially come from iPCBs that are present in consumer products including some pigments used in inks and paints (WA DOE, November 2016; Dilks, D., 2019; Guo et al 2014; Hu et al, 2010, NGC, 2018; NGC, June 2019; NGC, Feb 2019; WA DOE, Revised July 2021; WA DOE, November 2016)).

The Department of Ecology, Washington State, tested a selection of printing inks and found detectable levels of iPCBs (WA DOE, July 2022).

It has been found that effluent from recycling of printed paper has a higher level of PCBs than required wastewater limits (Inland Empire Paper Company PCB Fact Sheet, 2016).

This project has been created by convening a team of pigment, printing ink, and printing experts to use the ChemFORWARD Pigment Resource (ChemFORWARD, accessed 2023) to develop printing inks with no or ultra-low levels of iPCBs that have the required printing ink specifications and cost structure. Appendix A includes the phase 1 proposal to pilot the inadvertent PCB (iPCB) Pigment Resource that was approved by the Spokane River Regional Toxics Task Force in March of 2023.

This quality assurance project plan (QAPP) describes the process to:

- Test the iPCB content of four existing process inks (red, yellow, cyan and black) in use for newsprint within two project team printing facilities.
- Develop a no or ultra-low iPCB containing yellow process printing ink using alternative pigments identified through the ChemFORWARD Pigment Resource
- Determine the iPCB content of the no or ultra-low iPCB containing yellow process ink
- Evaluate the technical, printing, and cost performance of the no or ultra-low iPCB containing printing ink at one printing facility.

This Quality Assurance Project Plan (QAPP) describes the planned testing procedures for a pilot project that is designed to address the problem of inadvertent PCBs in printed paper by going "upstream" to the source. A proposal was developed and approved by the Spokane River Regional Toxics Task Force (SRRTTF) in the first quarter of 2023 to engage pigment and printing experts and to pilot a collaboration between an ink manufacturer, two printing facilities, and ChemFORWARD (project management). In this pilot, four color process inks currently used by

two different printers within the Spokane "recycle shed" will be tested for iPCBs. One of the printers has agreed to trial a reformulated ink with a focus on yellow. Yellow ink is anticipated to have the highest levels of iPCBs. One printer agreed to trial the replacement of their existing yellow process ink (presumed to contain inadvertently generated PCBs) with a reformulated ink that contains no, or ultra-low, levels of iPCBs because the pigments were manufactured without the use of chlorinated reagents and/or auxiliaries. The project will leverage information in the ChemFORWARD Pigment Resource, a database that was developed with funding from the Spokane Regional Toxics Task Force (SRRTTF), to identify pigment alternatives that are NOT likely to contain iPCBs.

The new ink formulation will be tested for iPCBs to determine that it does indeed contain no or ultra-low iPCB concentrations. Note that multiple replacement yellow ink formulations may be developed and tested for iPCBs as the project team works to meet performance requirements in the ink formulation via iterative development. Should iPCBs be found in an ink that contains a pigment that should theoretically contain no iPCBs, then components of the ink (other than the pigment) may be tested to determine the source of the iPCBs.

The ink will also be evaluated for printing performance characteristics and for its cost structure. The final report will include the type of ink used, the paper used, the type and age of the press, length of trial runs, and other aspects of the printing operations. Performance metrics will also be reported and will be based on the current performance metrics used by Lewiston.

This QAPP has been prepared for approval by the SRRTTF. The SRRTTF will be dissolved as of 30 June 2023 and at that time, the remaining funds at SRRTTF will be transferred to Ecology. It is anticipated that the original pilot proposal that was approved by the SRRTTF and this QAPP will be submitted to Ecology after the 30 June 2023 deadline. There was not enough time to prepare the QAPP and to complete the proposed project work and PCB testing prior to the dissolution of the SRRTTF. Even though the SRRTTF approved funding for the proposed pilot project, Ecology is not obligated to fund the project. It is anticipated that the QAPP will be reviewed by Ecology when it is submitted along with the proposal after 30 June 2023. At that time, further edits may be necessary.

3.2 Study area and surroundings

This project is not within a geographical area in the normal context of an Ecology project. This project will focus within industrial facilities as follows:

- Northwest Offset Printing (Northwest): a local commercial printer in the Spokane Valley area.
- Lewiston Morning Tribune (Lewiston); a publisher since 1892 serving print and paper packaging from north central Idaho and southeastern Washington, based in Spokane.
- Wikoff Color Corporation, an ink manufacturer based in Fort Mill, SC.

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Four color process inks (yellow, red, cyan and black) from Northwest and Lewiston will be collected and analyzed for iPCBs. Wikoff will develop a no or ultra-low iPCB ink alternative for the Lewiston yellow process ink and it will be analyzed for iPCB content. The project team will work with Lewiston to evaluate the no or ultra-low iPCB ink alternative for performance and cost as compared to the current Lewiston ink.

3.2.1 History of study area

The Department of Ecology, State of Washington has previously evaluated PCBs in Printing Inks (WA DOE, July 2022). It was found that the iPCB content ranged as follows:

- Black no result to 431ppb
- Cyan 0.9 to 547 ppb
- Magenta 7.7 to 298 ppb
- Yellow 101 to 40,200ppb

This project is novel as the development of no or ultra-low iPCB inks has not been attempted before.

3.2.2 Summary of previous studies and existing data

Washington State Department of Ecology's (Ecology's) Safer Products for Washington program identified PCB in printing inks as a priority chemical in 2020.

In 2021, Ecology's Product Testing program assessed the levels of PCBs in some products of the printing inks category. Twenty pigmented ink samples from five different companies were analyzed for 209 PCB congeners. Total PCB (tPCB) concentrations were calculated for 18 printing ink samples where 17 printing ink samples had detected levels of tPCBs, four had tPCBs below 1 ppb, four ranged from 1 to 100 ppb tPCBs, eight ranged from 100 to 1,000 ppb tPCBs, and one was above 1,000 ppb tPCBs (WA DOE, November 2021; WA DOE, July 2022 (authored by K. Trumbull). The results for each color was as follows:

- Black no result to 431ppb
- Cyan 0.9 to 547 ppb
- Magenta 7.7 to 298 ppb
- Yellow 101 to 40,200ppb

The Ecological and Toxicological Association of Dyes and Organic Pigments Manufacturers published the typical content of iPCBs within pigments (ETAD, 2022). Within the ETAD publication it was noted that pigment typically used in publication printing inks contained the following iPCB content:

- Black –(no data/ not reported)
- Cyan (phthalocyanine blue pigments) <2 ppm (<2000 ppb)
- Magenta (red pigments) <0.5 to 37 ppm (<500-37000 ppb)
- Yellow (diarylide yellows) <0.5 to 35 ppm (<500-35000 ppb)

ChemFORWARD has created a Pigments Resource (ChemFORWARD 2023), which is a free searchable dataset of nearly 400 pigments organized by chemical name, CAS#, color, and presence of chlorine. The tool can be used to find alternatives by avoiding those containing or manufactured with chlorine and potentially reducing the likelihood of containing iPCBs.

3.2.3 Parameters of interest and potential sources

The analytes of interest in this project are total incidental-polychlorinated biphenyls (iPCBs). iPCBs are permitted within the Toxic Substances Control Act (TSCA) up to a maximum allowable limit of 25 parts per million (ppm) on an annual average not to exceed 50 ppm. Inks have been found to contain iPCBs, mostly from (but likely not limited to) the pigments used to manufacture them.

This project will identify no or ultra-low iPCB containing pigments using the ChemFORWARD Pigment Resource (ChemFORWARD 2023) to be used to develop no or ultra-low iPCB printing inks as alternatives to the current inks in use.

3.2.4 Regulatory criteria or standards

Under the Toxic Substances Control Act (TSCA, 49 FR 28172), the concentration of inadvertently generated PCBs in products must have an annual average of less than 25 ppm, with a maximum of 50 ppm. It is expected the current inks being analyzed in this study, will be in full compliance with the TSCA regulatory limits.

3.3 Water quality impairment studies

Not applicable

3.4 Effectiveness monitoring studies

Not applicable

4.0 **Project Description**

This QAPP serves to outline an overall project to develop a no or ultra-low iPCB containing yellow printing process ink. The project will analyze the iPCB content in four process inks in use at two printers in the Spokane region, and subsequently develop a no or ultra-low iPCB containing ink to replace the yellow process ink for one printer with the objective to have the same or similar cost and performance as the existing ink. Depending on the PCB test results for the other process ink colors, additional work may be proposed as a follow-up to develop and replace formulations for the other process ink colors.

4.1 Project goals

The main goals of this study are to:

- Characterize and understand the iPCB content in 4 process inks in use at two different printers in Spokane
- Use the online ChemFORWARD Pigment Resource (ChemFORWARD 2023) in the pigment selection process
- Develop a no or ultra-low iPCB containing ink with similar (or same) cost and performance characteristics as the yellow ink currently in use.
 - No iPCB containing would mean PCBs are non-detect in the ink sample
 - Ultra-low iPCB containing is defined for the purposes of this project as <500 parts per billion (ppb). This number was selected as representing an upper limit for inks found to have the lowest levels of PCBs when tested by Ecology and reported in section 3.2.2 of this QAPP. Cyan (0.9 to 547 ppb) and black (no result to 431ppb) contained PCB levels considerably lower than those found in yellow ink (101 to 40,200ppb). As far as the project team knows, there is no established definition of ultra-low PCB levels to serve as precedent.
- Run print trials of the no or ultra-low iPCB containing ink to confirm its performance and effectiveness

4.2 Project objectives

In support of the project goals, the following objectives will be carried out:

- Analyze existing ink formulations for the presence of iPCBs for 4 base colors (black, cyan, red and yellow) from two printing facilities
- Identify performance requirements and acceptable cost criteria
- Identify alternative pigments that should have no or ultra-low iPCBs

- Formulate a new ink that meets the performance and cost targets using the recommended alternative pigments. (Note that multiple formulations may be trialed and tested in the process of creating an acceptable replacement ink.)
- Analyze the new ink formulation(s) for iPCBs
- Run print trials of the new no or ultra-low iPCB containing inks
- Compare the initial and revised formulations for cost and performance as described in section 7.5.2

4.3 Information needed and sources

No further background data is necessary

4.4 Tasks required

In order to achieve study objectives, the following tasks are required:

- Scheduling of meetings and discussions with project team
- Coordination of sample collection from Lewiston Morning Tribune and Northwest Offset Printing
- Submission of ink samples for laboratory analysis of PCB congeners by EPA Method 1668C
- Analysis of samples for PCB congeners
- Verification of PCB data
- Validation of data for usability
- Selection of no or ultra-low iPCB containing pigments
- Formulation of ink(s) with no or ultra-low iPCB containing pigments
- Evaluation of cost and performance characteristics of the new no or ultra-low iPCB containing ink(s) compared to the current in use inks at laboratory level
- Analysis of reformulated ink(s) for PCB congeners
- Evaluation of cost and performance characteristics of new no or ultra-low iPCB containing ink(s) compared to the current in use ink in full print trials
- Preparation of and issuing final report

4.5 Systematic planning process

This QAPP establishes a suitable systemic planning process.

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Table 1 shows the responsibilities of those who will be involved in this project. The source of funding is to be determined.

| Staff | Title | Responsibilities |
|---|--|---|
| tbd | Project Funder tbd | Manage contracts: review and approve project specifications. Ensure project is completed in timely manner |
| Lauren Heine ChemFORWARD Phone: 360-220-2069 (mobile) | Project Manager, Principal Investigator | Oversee and coordinate the project. Responsible for completion of the QAPP as well as draft and final reports. Ensure work is done in accordance with the QAPP. |
| Mark Vincent Chroma Specialty Chemicals Phone: 416-702-9984 | Pigment Expert and Project Advisor | Advise on iPCB testing of the materials and advise on no or ultra-low iPCB alternatives. Draft the QAPP and support writing of the final report |
| Grace Manarang-Pena Chroma Specialty Chemicals Phone: 289-830-4232 | Pigment and Regulatory Expert | Draft the QAPP. Coordinate iPCB sample testing with the laboratory. Review iPCB test data to ensure that it is in accordance with the QAPP. |
| Anne Stephens Wikoff Color Corporation Phone: 1 803.835.8305 (office) | Ink Development | Responsible for development of no or ultra-low iPCB inks |
| Nathan Alford Lewiston Morning Tribune/Revolve Print and Pack Phone: 208-848-2208 | Print Trials | Provide samples of inks that are currently used. Responsible for completion of print trials with no or ultra-low iPCB inks developed by Wikoff |
| Doug Krapas Inland Empire Paper Phone: (509) 924-1911 (office) | NEP Quality Coordinator; Chair TSCA Workgroup of the SRRTTF | Chaired the TSCA Workgroup of the Spokane River Regional Toxics Task Force |
| Mr. Riff Mattre Northwest Offset Printing Office 509-459-5283 | WA Printer | Provide samples of inks that are currently used |
| Arati Kaza Department of Ecology Phone: 360-407-6964 | Quality Assurance Officer | Review and approve the QAPP for Ecology once it is submitted with the proposal after 30 June 2023. |

5.2 Special training and certifications

No Special training is necessary. The key personnel are highly trained and experienced within their subject matter.

5.3 Organization chart

The lines of reporting for the organizations in the project are shown in the organization chart (Figure 1). Currently ChemFORWARD is contracted for project management by the SRRTTF and works directly with Doug Krapas of Inland Empire Paper, who chairs the SRRTTF TSCA Workgroup. The individuals currently identified in project funding and project management roles may change once the SRRTTF is dissolved and the proposal is submitted for approval and funding elsewhere.

The project management team coordinates communication and the transfer of inks to Eurofins in Sacramento, California for testing. The project manager also coordinates and/or monitors the transfer of inks between Wikoff and Lewiston for performance trials.

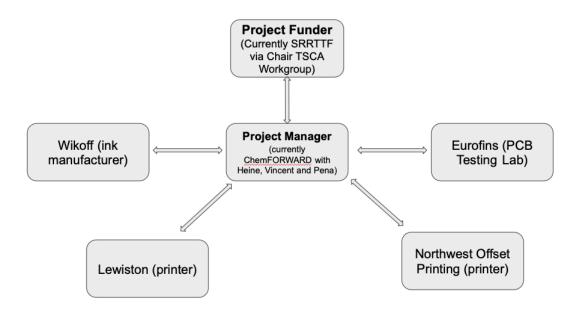


Figure 1. Project Organization Chart

5.4 Proposed project schedule

Start and end dates for the major project activities are provided below in Tables 2 assuming a start date of July 2023. The dates will be revised once project funding is secured.

| Task | Start Date Due Date | | Lead Staff |
|---|---------------------|----------------|---|
| Baseline iPCB Testing | | | |
| Testing of Lewiston base inks | July 2023 | August 2023 | Grace Manarang-Pena |
| Testing of Northwest base inks | July 2023 | August 2023 | Grace Manarang-Pena |
| Low/no iPCB ink Development | | | |
| Identify low or no iPCB yellow pigments | July 2023 | July 2023 | Mark Vincent Anne Stephens |
| Low/no iPCB yellow ink Development | July 2023 | September 2023 | Anne Stephens |
| Evaluation of low/no iPCB yellow ink | August 2023 | October 2023 | Nathan Alford |
| Scale up low/no iPCB yellow ink | October 2023 | November 2023 | Anne Stephens |
| Yellow press trials | November 2023 | December 2023 | Nathan Alford |
| iPCB testing of new inks | July 2023 | December 2023 | Grace Manarang-Pena |
| Final Report | | | |
| Draft report to Task force | December 2023 | February 2024 | Lauren Heine Mark Vincent Grace Manarang-Pena |
| Final report | April 2024 | April 2024 | Lauren Heine |

5.5 Budget and funding

Funding for the creation of this QAPP was provided by the Spokane River Regional Toxics Task Force (SRRTTF) with a completion date before 30 June 2023.

Funding in support of the project to Pilot the Inadvertent PCB (iPCB) Pigment Resource to Develop no or ultra-low iPCB Inks for Printing was approved by the SRRTTF in the first half of 2023. However, there was insufficient time to complete the project. Therefore, the pilot project proposal will need to be submitted to a different funder (ideally Ecology) after the SRRTTF is dissolved on 30 June 2023.

The budget for the pilot project that was initially submitted to the SRRTTF is broken down in Table 3 below. It includes funding for 33 PCB analyses.

Table 3. Project budget and funding

| Task | Team Lead | Rate | | Rate Hours or Qty | | rs Labor | | Raw Material and Testing Costs (Not to exceed) | | In-kind Contribution | | Total (\$) | |
|--|-----------------------------------|------|-----|----------------------|----|----------|----|--|----|-------------------------|----|------------|--|
| Project Management | ChemFORWARD/Heine | | | | | | | | | | | | |
| Lauren Heine | | \$ | 200 | 72 | \$ | 14,400 | | | | | \$ | 14,400.00 | |
| PCB Testing | Eurofins Testing Lab | \$ | 950 | 33 | \$ | - | \$ | 31,350.00 | | | \$ | 31,350.00 | |
| Research & Development | ChemFORWARD/Vincent | | | | | | | | | | | | |
| Mark Vincent | | \$ | 200 | 60 | \$ | 12,000 | | | | | \$ | 12,000 | |
| Lauren Heine | | \$ | 200 | 20 | \$ | 4,000 | | | | | \$ | 4,000 | |
| Formulation R&D and printing labor (in-kind) | Wikoff, Chroma SC and Lewiston | \$ | 200 | 170 | | | | | \$ | 34,000.00 | \$ | - | |
| Raw Materials | | | | | | | | | | | | | |
| (pigments, inks, and pilot material runs) | | | | | | | \$ | 50,000.00 | | | \$ | 50,000.00 | |
| Reporting | | | | | | | | | | | | | |
| Lauren Heine | | \$ | 200 | 10 | \$ | 2,000 | | | | | \$ | 2,000.00 | |
| Mark Vincent | | \$ | 200 | 6 | \$ | 1,200 | | | | | \$ | 1,200.00 | |
| Subtotal | | | | | | | | | \$ | 34,000.00 | \$ | 114,950.00 | |
| HBN Administrative Fee | | 1 | 10% | | | | | | | | \$ | 11,495.00 | |
| Total cost of proposed work to SRRTTF | | | | | | | | | | | \$ | 126,445.00 | |
| Total value with in-kind contribution | | | | | | | | | | | \$ | 160,445.00 | |

iPCB testing will be carried out by Eurofins Testing Northwest, LLC (Eurofins). The number of samples for each phase of the project is detailed in Table 4.

Table 4. iPCB samples for testing

| Samples | Number of Samples | Number of QA Samples | Total Number of Samples | Cost Per Sample (\$) | Lab Subtotal (\$) |
|--|----------------------|----------------------------|-------------------------------|----------------------------|-------------------------|
| Lewiston base inks | 4 | 4 | 8 | 950 | 7,600 |
| Northwest base inks | 4 | 4 | 8 | 950 | 7,600 |
| Wikoff Developmental inks | 15 | 0 | 15 | 950 | 14,250 |
| Testing of final ink for press trials | 1 | 1 | 2 | 950 | 1,900 |
| Total | 24 | 9 | 33 | 950 | 31,350 |

6.0 Quality Objectives

6.1 Data quality objectives

The main data quality objective (DQO) for this project is to obtain results of documented accuracy of PCB content for 33 ink samples representative of current ink formulations,

developmental inks and reformulated inks. The EPA method 1668C will be used to identify and determine the concentrations of the congeners present. The data quality will be evaluated against the measurement quality objectives (MQOs) for precision, bias and sensitivity.

6.2 Measurement quality objectives

6.2.1 Targets for precision, bias, and sensitivity

In order to obtain data of sufficient quality to access the concentration and sum of the 209 congeners, the MQOs, expressed in terms of acceptable precision, bias, and sensitivity, are shown in Table 5.

| | Precision | | Bias | | Sensitivity |
|----------------------|--|-------------------------------|---|-------------------------------------|-----------------|
| Analyte⁺ | Laboratory Control Standard Duplicates (RPD) | Sample Duplicates (RPD) | Laboratory Control Standards (Recovery)* | Labeled compounds (Recovery)* | Reporting Limit |
| 209 PCB Congeners | ±25% | ±50% | 60-135% | 5-145% | 2 ppb |

Table 5. Measurement quality objectives for laboratory analyses

⁺Target analytes for 209 PCB congeners are listed in Appendix A Table A-1

*Excerpt shown in Appendix Table A-2. Complete listing of Ongoing precision and recovery (OPR) Standards and labeled compounds recovery are in Table 6 of EPA Method 1668C

RPD = Relative Percent Difference

ppb = parts per billion

6.2.1.1 Precision

Precision measures the variability among replicate measurements due to random error.

The laboratory analysis precision will be based on the assessment of laboratory duplicate samples as outlined in Table 5. In addition, duplicate samples will be collected and analyzed at each stage of this project.

6.2.1.2 Bias

The difference between the sample mean and the true value is known as bias. In this study, the laboratory analysis bias will be assessed through the MQOs for laboratory control standards.

6.2.1.3 Sensitivity

The capability of a method to detect a substance above the background level is a measure of sensitivity. The detection limit or reporting limit of PCB congeners for this study is noted in Table 5.

6.2.2 Targets for comparability, representativeness, and completeness

6.2.2.1 Comparability

Standardized sample collection procedures will be implemented to ensure comparability. Where possible samples in original unopened product containers will be submitted to the analytical labs for analysis. For aliquoted samples, the inks will be collected and stored in amber wide mouth jars with Teflon lids provided by Eurofins.

The approved Eurofins laboratory SOP WS-ID-0013 for PCB analysis by Method 1668C and PCB sample preparation for Method 1668C will be used for this study. Eurofins is an accredited laboratory qualified to conduct PCB analysis according to EPA Method 1668C.

6.2.2.2 Representativeness

The base inks collected for this study will be representative of those commercially available and used by local printers, namely Lewiston and Northwest.

The developmental inks will be formulated with commercially available raw materials including pigments from approved/qualified suppliers. Performance testing for the inks at Lewiston will be conducted by trained operators using standard procedures. Press trials will be run at Lewiston's local printing facility using standard procedures and equipment.

6.2.2.3 Completeness

This project will be considered complete if developed ink meets the targeted iPCB and ink performance levels.

6.3 Acceptance criteria for quality of existing data

Not applicable to this study.

6.4 Model quality objectives -NA

Not applicable to this study.

7.0 Study Design

7.1 Study boundaries

This study does not involve fieldwork or field activities and will take place within printing operations, laboratory, and industrial facilities. There are no geographical study boundaries. The project involves collecting ink samples from Lewiston Morning Tribune/Revolve Print and Pack (Lewiston), Lewiston, Idaho as well as Northwest Offset Printing (Northwest), Spokane Valley, Washington and having them tested within Eurofin Laboratories. Development of the low/no iPCB inks will be carried out by Wikoff Color Corporation in Fort Mill, North Carolina. The low/no iPCB inks will also be tested by Eurofin Laboratories.

7.2 Field data collection

Not applicable.

7.2.1 Sampling locations and frequency

Sampling of two sets of four color process inks (yellow, red, cyan and black) will be collected from Lewiston Morning Tribune/Revolve Print and Pack, Lewiston, Idaho as well as Northwest Offset Printing, Spokane Valley, Washington. These samples will be analyzed for iPCB content. For accuracy the samples will be tested in duplicate.

For this pilot project, focus will be on developing a no or ultra-low iPCB yellow ink. During the developmental phase, up to 15 developmental samples would be anticipated to be tested for iPCB content. This may also include selected pigments and ink components should unexpected iPCB results be received.

If a developmental ink is approved by Lewiston for press trials, this final ink will be scaled up and tested for iPCB content in duplicate.

7.2.2 Field parameters and laboratory analytes to be measured

This project will focus on iPCBs. The parameters are described in Section 6.2.

7.3 Modeling and analysis design

Not applicable.

7.4 Assumptions of study design

The assumptions associated with the study design are:

- 1. The collected four color process inks will contain iPCBs
- 2. The yellow process inks will contain the highest or one of the highest levels of iPCBs
- 3. Development of a no or ultra-low iPCB yellow ink is possible with no or ultra-low iPCB pigments

- 4. The inks are highly customized for the type of paper and type and age of press and results may not be directly transferable to other printing applications.
- 5. Development of a no or ultra-lowiPCB yellow ink with the appropriate printing performance and cost characteristics is achievable
- 6. Eurofins can accurately and consistently test iPCB content in inks

7.5 Possible challenges and contingencies

7.5.1 Logistical problems

No major logistical problems are expected within the timeframe of the project.

7.5.2 Practical constraints

Practical constraints within the project will be whether it is possible to develop a no or ultra-low iPCB yellow ink able to meet the performance and cost requirements. The project team envisions an iterative process whereby initial formulation of the reformulated yellow process ink will lead to initial performance testing. If the ink passes the initial performance tests, then it may be tested for PCBs before making further modifications to the formulation. This is likely to be followed by more extensive performance testing before final PCB testing. There will be new learnings as the project progresses and decisions will be made as each ink sample is evaluated for ink performance as well as iPCB content.

Performance: The evaluation of performance is based on the performance needs of the printer which are specific to the newsprint and the press that are used. Initial performance tests are expected to be less extensive than final performance evaluations during ink development. Below are some key considerations for performance evaluation. However, we are constrained by the performance considerations and criteria that are actually used by the printer. All test considerations and conditions used to evaluate performance will be captured in the final report.

Key considerations for comparing the performance of the incumbent to the substitute ink with respect to the printing trials may include:

- Duration: The comparison print runs should be run over significant durations, at least 3-4 hours, as some of the issues will come to light as the press is running. It is recommended that the press speed should be at a minimum of 80% of the rated speed. In addition, the following information for the press is recommended for measurement and recording:
 - a. Ink roller type and hardness
 - b. Blanket type and hardness
 - c. Press speed for both inks
 - d. Press settings for ink feed
 - e. Press settings for fountain solution delivery and if any adjustments are needed for the fountain solution mix ratio.
 - f. Notation of any misting that occurs.
 - g. Ink consumption to determine mileage.
 - h. Any other adjustments made during the production run.

- 2. Prepress Software: Settings and any changes made to the prepress software for imaging the plates for the test inks will be identified and recorded.
- 3. Ink Lab Tests: the following lab tests on the ink are recommended:
 - a. Rub resistance
 - b. Drying time and other characteristics
 - c. Density measurement using a Little Joe
 - d. Ink Draw Down
 - e. Water Pick Up to measure emulsification.
 - f. Tack
 - g. Viscosity

Cost: With respect to cost, the project team will prepare a summary of the relative cost. We expect that the ink supplier and printer are unwilling to share exact costs based on confidential business information. Therefore, we will report a metric for comparative cost, i.e., the cost of the newly formulated alternative relative to the cost of the current ink formulation.

7.5.3 Schedule limitations

The major schedule limitation is the turnaround for iPCB testing. It is currently estimated at 2-3 weeks but can take up to 8 weeks to receive results. The lab has committed to 2-3 weeks for this project. This will be managed by the project team to prevent time slippage.

No other major schedule limitation is expected.

8.0 Field Procedures

8.1 Invasive species evaluation

Not applicable to this study.

8.2 Measurement and sampling procedures

Lewiston and Northwest will provide base inks selected from their approved suppliers.

Wikoff will develop inks using raw materials from qualified suppliers. Raw material suppliers are audited and approved based on Wikoff's quality management system ISO 9001:2015 (ISO certified since 2010).

All ink samples with unique product identifiers will be submitted to the analytical laboratory. Samples to be sent directly to Eurofins for sample preparation as per Method 1668C (SOP No. WS-IDP-0013, Rev.4.3)

8.3 Containers, preservation methods, holding times

Table 6 summarizes the sample requirements. EPA Method 1668C has not established maximum holding times or preservation methods for liquids including printing inks.

Where possible samples in original unopened product containers will be submitted to the analytical labs for analysis. For aliquoted samples, the inks will be collected and stored in amber wide mouth jars with Teflon lids.

| Parameter | Matrix | Minimum Quantity Required | Container | Sample Storage and Preservation | Holding Time* |
|---------------|-----------------|---------------------------------|---|---|------------------|
| PCB Congeners | Printing Ink | 5-10 grams | 4 oz Amber glass jar or original unopened container | Minimize exposure to light, keep at ambient temperature | 1 year |

Table 6. Sample containers, preservation, and holding times.

8.4 Equipment decontamination

High levels of contaminants are not anticipated during the ink sample collection. Base ink and reformulated inks are not anticipated to be exposed to contaminants that will significantly impact PCB analysis.No decontamination necessary is for sample preparation for transport to the laboratory.

The laboratory equipment preparation and decontamination will follow the protocol as outlined in Eurofin SOP No WS-IDP-0013, Rev.4.3

Laboratory method blanks will be run to demonstrate that the solvents, reagents, glassware and other sample processing hardware are free from interference that can cause misinterpretation of chromatographic data. PVC gloves will not be used by the analysts and reuse of glassware will be minimized.

Clean up techniques are outlined within the SOP. However additional clean ups steps may be required to achieve lower detection limits.

8.5 Sample ID

Each ink sample will be assigned a unique product identifier that will incorporate:

- Project stage: 1 = base ink, 2 = developmental ink, 3= trial ink
- Supplier code where L= Lewiston, N=Northwest, W=Wikoff
- Colour Code: C= Cyan, Y=Yellow, M=Magenta, B=Black

• Sample Number: 1, 2 etc

8.6 Chain of custody

Chain of custody will be maintained for all the samples for this study. Lewiston, Northwest and Wikoff will use the Eurofin chain of custody for the transport of samples from their facility to the laboratory.

8.7 Field log requirements

Sample selection and collection will be recorded in the Product Documentation Log.

The following information will be recorded:

- Name and location of sampling facility
- Personnel responsible for sample collection
- Date, time, location, ID, and description of each sample
- Sampling conditions
- Relevant SOP procedures
- Changes or deviations from the QAPP or SOPs
- Unusual circumstances that might affect interpretation of results

8.8 Other activities

Necessary activities are detailed in other sections of this QAPP.

9.0 Laboratory Procedures

9.1 Lab procedures table

Table 7 summarizes the samples, methods, reporting limit and expected range of results for the PCB congeners under US EPA Method 1668C. This procedure includes the extraction, analyte specific clean up and HRGC/HRMS analysis techniques.

The samples will be sent to Eurofins. The preparation of the final extract is outlined in SOP No.WS-IDP-0013 Rev.4.3. Five isotopically (13 C12) labeled internal standards used to determine the percent recoveries.

One to two microliters of the final concentrated extract are ingested in the HRGC/HRMS.

The identification of the PCB congeners is based on their elution time measured in the routine calibration standard and the simultaneous detection of the two most abundant ions in the molecular ion isotopic cluster.

When the ¹³C-labeled isotope dilution analyte is available, the PCB identification is based on their elution time compared to the corresponding labeled isotope dilution analyte and the simultaneous detection of the two most abundant ions in the molecular ion isotopic cluster.

The Project Manager will be notified of any significant deviations to the SOP procedures and determine the appropriate course of action.

| Analyte | Sample Matrix | Samples (Number/ Arrival Date) | Expected Range of Results | Detection or Reporting Limit | Sample Prep Method | Analytical (Instrumental) Method |
|----------------------|------------------|---|---------------------------------|------------------------------------|---|--|
| 209 PCB Congeners | Printing ink | 33 | Up to 1 ppm | 2000 ppg/g | EPA 1668C; Lab SOP No.WS-IDP- 0013, Rev.4.3 | EPA 1668C; Lab SOP No.WS-ID-0013, Rev.4.9 |

Table 7. Measurement methods (laboratory).

9.2 Sample preparation method(s)

When unopened ink containers are not viable for sampling, an aliquot of the inks will be collected as follows:

- Wear appropriate personal protective equipment (PPE) e.g., gloves, eyewear, and lab coat, while collecting product samples.
- Vigorously mix product in unopened original container. Open the lid of the product container.
- Open the lid of clean sample jar with clean gloves and pour a well-mixed aliquot of the
- original product into a clean jar.
- Replace the lid of the jar without touching the inside of the jar or lid.
- Ensure that the aliquot poured from the original container does not run down the side of the container but is a clean pour directly from the original container into a clean sample jar.

Lab sample preparation for analysis will adhere to **Eurofins Lab SOP No.WS-IDP-0013, Rev.4.3** (internal test method). An aliquot is spiked with a solution containing 27 isotopically ¹³C-labeled PCBs as described in the US EPA Method 1668C Guidance and listed in <u>Appendix B</u> prior to extraction (US EPA, 2010). Surrogate standards are spiked into the extract prior to performing clean ups.

The preparation of the final extract for the instrumental analysis is accomplished by adding 5 isotopically (¹³C12) labeled internal standards. After internal standards are added and the extract is concentrated to 20uL, the extract is then analyzed according to SOP WS-ID-0013.

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Quantitation analysis is carried out using isotope dilution against a 5-point calibration (for Toxic and LOC PCBs) or internal standard technique using a single calibration point for all other congers.

9.3 Special method requirements

No special method requirements are anticipated. Any modification to the EPA Method 1668C will be reviewed with the Project Manager and outlined in the analytical laboratory data package.

9.4 Laboratories accredited for methods

Eurofins Environment Testing Northwest, LLC is an Ecology-accredited laboratory for PCB congener analysis by EPA Method 1668C. Eurofins will conduct the PCB analysis for this study.

10.0 Quality Control Procedures

10.1 Table of field and laboratory quality control

Table 8 presents each type of QC sample that will be used to evaluate the quality and usability of the results based on the MQO listed in Section 6.2.

One method blank will be extracted at each stage of the project: base ink, development ink and press trial ink. The method blank is an aliquot of the reference matrix processed in the same manner and at the same time as the associated samples.

A laboratory control sample (LCS) also will be extracted at each stage of the study. The LCS is an aliquot of laboratory matrix spiked with 100 uL of Isotope Dilution Analyte fortification solution and 100 uL of Target Analyte Standard fortification solution. The LCS will be processed in the same manner and at the same time as the associated samples.

Table 8. Quality control samples, types, and frequency.

| Parameter | Samples | Laboratory Method Blanks | Laboratory Control Sample | Laboratory Control Sample Duplicate |
|----------------------|---------------------|-----------------------------|------------------------------|---|
| 209 PCB Congeners | 16 Process Inks | 1/batch | 1/batch | 1/batch |
| 209 PCB Congeners | 15 Development Inks | 1/batch | 1/batch | 1/batch |

| 209 PCB Congeners 2 Press Trial Inks | 1/batch | 1/batch | 1/batch | |
|---|---------|---------|---------|--|
|---|---------|---------|---------|--|

10.2 Corrective action processes

The laboratory packages will document any departures from the analytical method and QC criteria along with results that do not meet the MQOs. These deviations will be included in the final report along with any deviations from this QAPP.

The appropriate corrective actions which may include re-sampling, re-testing or rejection of data will be determined by the project manager

11.0 Data Management Procedures

11.1 Data recording and reporting requirements

All data including the analytical reports from Eurofins Environment Testing Northwest, LLC. will be included in or appended to the overall project report that will be written by the project management team and provided to the funder. Comparative PCB testing results and the performance and cost analyses will also be included in the final project report. The testing results from the currently used individual inks and the reformulated trial inks are not intended for uploading to US EPA or Ecology websites.

11.2 Laboratory data package requirements

Two analytical report formats will be provided by Eurofins Environment Testing Northwest, LLC. The final report in PDF format and standard electronic data deliverable (EDD) will be provided via e-mail or web portal. The Tier 3 Level data package will include summary information, results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses. Tier 3 also includes calibration information. Note that the data package requested may be raised to Tier 4 to ensure access to all raw supporting data, pending funder requirements.

Corrective actions, changes to the requested analytical method and glossary will also be included in the data package.

11.3 Electronic transfer requirements

Data packages will be in PDF format and/or alternative format approved by the Project Manager.

11.4 Data upload procedures

Not applicable to this study. Data for this study will be stored according to Section 11.1.

11.5 Model information management

Not applicable to this study.

12.0 Audits and Reports

12.1 Audits

The analytical lab is subject to performance and system audits as part of their routine procedures. The Project Management team will ensure that the study adheres to the QAPP.

12.2 Responsible personnel

The project management team will conduct an audit of the study processes including product acquisition, product documentation and sample screening, sample processing, chain of custody and adherence to product testing.

12.3 Frequency and distribution of reports

Short format reports summarizing data and findings at each stage of the study will be published. The final report will encompass:

- Study overview
- Goals and objectives of the study
- General description of products sampled
- Discussion of methods, test results, data quality and significance of any problems encountered
- Summary of tables and graphs of laboratory data
- Summary of performance and cost evaluations
- Conclusions and recommendations

12.4 Responsibility for reports

The project manager will be the lead responsible for the final published report.

13.0 Data Verification

13.1 Field data verification, requirements, and responsibilities

The project manager will conduct the final review of the ink sampling, submissions to the analytical laboratory and data package.

13.2 Laboratory data verification

The project manager will conduct a review to verify adherence to the protocols in this QAPP and QC requirements of EPA Method 1668C. This will review will evaluate:

- Methods and protocols specified in the study
- All calibrations, QC checks, and intermediate calculations performed for all samples
- Data for consistency, accuracy and completeness

A report on the overall assessment of MQO, data quality and usability will be included in the study report.

13.3 Validation requirements, if necessary

External data validation is not anticipated to be required for this study.

13.4 Model quality assessment

Not applicable

14.0 Data Quality (Usability) Assessment

14.1 Process for determining project objectives were met

The team has two major objectives:

- 1. Test existing printing inks in use
- 2. Develop ultra-low or no iPCB printing inks with acceptable performance characteristics

The Project Manager and Pigment & Regulatory Expert will review the analytical data and whether it meets the project MQOs. The data will either be accepted, accepted with qualification, or rejected. The Project Manager and Pigment & Regulatory Expert will determine if any samples need to be reanalysed should any results not meet the MQOs.

The Ink Development and Print Trial teams will review the ink performance and cost characteristics and determine if the newly developed ultra-low or no iPCB inks have met the desired performance objectives. If rejected, they will determine if redevelopment is required or possible.

14.2 Treatment of non-detects

EPA Method 1668C allows for low-level detection of PCB congeners. However, PCB congeners may be present in laboratory method blanks at higher concentrations than the detection limit. Congener results that are less than five times the detected method blank concentration will be censored and qualified as non-detects, "U" or "UJ".

PCB congener results censored as non-detects will be:

- qualified as "U" (the analyte was not detected at or above the reported concentration) when the concentration is less than five times the detected method blank concentration and greater than the limit of quantitation (LOQ).
- qualified as "UJ" (the analyte was not detected at or above the estimated concentration) when the concentration is less than five times the detected method blank concentration and less than the LOQ but greater than the estimated detection limit (EDL).
- qualified as "UJ" when the concentration of a tentatively identified PCB congener, qualified as "NJ," is less than five times the detected method blank concentration and less than the LOQ but greater than the EDL.

Non-detected congener results will not be included in calculation of total PCBs, the sum of PCB congeners in the sample

14.3 Data analysis and presentation methods

PCB congener results below the LOQ and above the EDL will be qualified "J" (indicating that the analyte was positively identified and the concentration is an estimate). PCB congener results less than five times the method blank contamination will be censored as non-detects (section 14.2).

Total PCBs will be calculated from PCB congener results as the sum of PCB congeners in the sample and include only detected congener results that are qualified "J," as estimates, and detected congeners without qualification. Non-detected congener results and congener results qualified as "NJ" (indicating the analyte has been tentatively identified and the concentration is an estimate), will not be included in the total PCB sum of congeners. Total PCB calculations will be qualified "J" when 10% or more of the detected congener concentration results are qualified "J."

In addition, PCB congener profiles will be examined and discussed. A summary of the data will be presented in the final report. Results will be displayed in tables, graphs, and/or charts.

14.4 Sampling design evaluation

The number and type of samples collected and tested should be sufficient to meet the objectives of the specific study event. The results of the study may lead to future study events with a larger sample size and/or a wider variety of products. Additional study events will be described in a QAPP addendum.

14.5 Documentation of assessment

Documentation of assessment will occur in the final report (see Section 12).

15.0 References

- 1. ChemFORWARD. Accessed 2023. Inadvertent PCB (iPCB) Pigment Resource home page. LINK
- 2. ChemFORWARD. Accessed 2023. Inadvertent PCB (iPCB) Pigment Resource database. LINK
- Dilks. D.W. LimnoTech, Inc. 2019. PCBs in our Watershed, PCBs in Products, and TSCA Exclusions: Putting it All Together. Presentation to the Spokane River Regional Toxics Task Force. <u>LINK</u>
- ETAD Position on the Presence of Unintentional Trace PCBs in Some Organic Pigments in the Context of Regulation (EU) 2019/1021 (POPs recast Regulation), Version 2, June 15th 2022. <u>LINK</u>
- Guo, J., Capozzi, S., Kraeutler, T. and L. Rodenburg. 2014. Global Distribution and Local Impacts of Inadvertently Generated Polychlorinated Biphenyls in Pigments. Environmental Science and Technology, 48: 8573-8580. <u>LINK</u>
- 6. Hu, D. and Hornbuckle, K.C. 2010. Inadvertent Polychlorinated Biphenyls in Commercial Paint Pigments. Environmental Science and Technology, 44: 2822-2827. <u>LINK</u>
- 7. Inland Empire Paper (IEP). 2016. Inland Empire Paper Company PCB Fact Sheet. LINK
- 8. Northwest Green Chemistry (NGC). October 2018. Inadvertent PCBs in Pigments: Market Innovation for a Circular Economy. Final Report. Prepared for the Spokane River Regional Toxics Task Force. <u>LINK</u>
- Northwest Green Chemistry. Feb. 2019. The Potential for Generating Inadvertent PCBs through TiO₂ Manufacturing Using the Chloride Process. Prepared for the Spokane River Regional Toxics Task Force. <u>LINK</u>
- Northwest Green Chemistry (NGC). June 2019. Pigments and Inadvertent Polychlorinated Biphenyls (iPCBs): Advancing No and Low iPCB Pigments for Newsprint, and Paper and Paperboard Packaging. Prepared for the Spokane River Regional Toxics Task Force. <u>LINK</u>
- 11. Spokane River Regional Toxics Task Force (SRRTTF) website. Accessed 2023. LINK
- 12. United States Environmental Protection Agency (US EPA). April 2010. Method 1668C Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (Table 1). LINK
- Washington Department of Ecology (WA DOE) November 2021. Quality Assurance Project Plan: PCBs in Washington State Products. Authored by Kari Trumbull. Publication 21-03-121. <u>LINK</u>
- 14. Washington Department of Ecology (WA DOE). July 2022. Authored by Kari Trumbull. PCBs in Washington State Products: Printing Inks, Publication No. 22-03-001. <u>LINK</u>
- 15. Washington Department of Ecology (WA DOE). Revised July 2021. Quality Assurance Project Plan. Polychlorinated Biphenyls (PCBs) in General Consumer Products. Publication No. 13-04-008. <u>LINK</u>
- 16. Washington Department of Ecology (WA DOE). November 2016. Polychlorinated Biphenyls in Consumer Products. Publication No. 16-04-014. LINK

16.0 Appendices

Appendix A. Phase 1 Proposal to Pilot the Inadvertent PCB (iPCB) Pigment Resource Approved by the Spokane River Regional Toxics Task Force on 2023_03_15

ChemFORWARD is pleased to submit this proposal to the Spokane River Regional Toxics Task Force (SRRTTF) to pilot the use of the online ChemFORWARD Pigment Resource (LINK) to replace ink formulations containing inadvertently generated PCBs (iPCBs) with those that should contain no, or ultra-low, concentrations of iPCBs from pigments. The ChemFORWARD Pigment Resource identifies pigments that are manufactured without the use of chlorinated solvents and that do not contain chlorine in their molecular structure. This work is proposed in phases and builds on the prior efforts of ChemFORWARD for the SRRTTF.

ChemFORWARD has convened a team of experts and practitioners including a pigment supplier, an ink manufacturer, a paper manufacturer and publishers of newsprint and paper packaging who will collaborate to:

- 1. Test currently used ink formulations for the presence of iPCBs,
- 2. Identify performance requirements and acceptable cost criteria for ink formulations,
- 3. Identify alternative pigments that should not contain iPCBs or have much lower levels,
- 4. Formulate new ink formulations that use the recommended alternative no/ultra-low PCB pigments and that meet performance and cost targets,
- 5. Test the new ink formulations for iPCBs, and
- 6. Compare the initial and revised formulations for cost and performance.

Partners: (See appendix for more detailed description of partners):

- Lewiston Morning Tribune/Revolve Print and Pack (Lewiston) (in-kind)
- Chroma Specialty Chemicals (Chroma team; i.e., Mark Vincent and Grace Manarang-Pena, subcontractors)
- Inland Empire Paper (in-kind)
- Wikoff Color Corporation
 - o In-kind contribution of time
 - o Materials in budget
- Northwest Offset Printing (Local WA publisher and sister company to Inland Empire Paper; will provide additional samples for testing)
- Testing Lab (Eurofins)
- ChemFORWARD (project lead)

Scope of Work

1. Project Management

Project management will be performed by Lauren Heine for ChemFORWARD. Project management includes setting up weekly calls with Mark Vincent and Grace Manarang-Pena (Chroma team) and others from the broader project team as needed. It includes coordinating and/or tracking sample testing, ink reformulations, and performance testing results. The Chroma team will help establish performance requirements, identify alternative pigments, review new formulations, and help to evaluate performance results

2. Test currently used ink formulations for the presence of iPCBs

Lewiston Morning Tribune (Lewiston) currently uses four base colors (yellow, red, cyan, and black) to print newsprint and paper packaging. A sample of each color will be tested using EPA method 1668. Samples will be run in at least duplicate to capture variability. A second printing company, Northwest Offset Printing will join the pilot, and provide samples of their inks for PCB testing. Northwest Offset Printing will also test samples of four base colors (in duplicate). (*Note: Northwest Offset Printing is participating ONLY in the testing of PCBs in their inks and not in the ink reformulation part of this project*.) A total of 33 samples are expected to be tested during this project. The PCB testing budget is set at a "not to exceed" limit.

3. Identify performance requirements and acceptable cost criteria (Lewiston, Wikoff and Chroma)

The ChemFORWARD research team will work with Lewiston and Wikoff Color Corporation to specify the performance requirements and to set a target cost for viable alternative ink formulations. A sample (kit) of the yellow ink used by Lewiston will be sent to Wikoff Color Corporation to help them establish a benchmark for performance and cost for yellow ink.

4. Identify alternative pigments that should not contain iPCBs

The ChemFORWARD research team (Chroma) will use the ChemFORWARD Pigment Resource and will recommend one or more alternative pigments that can be used to substitute for the pigments currently used in Lewiston yellow ink formulations.

5. Formulate new ink formulations that use the recommended alternative pigments and that meet performance and cost targets

The team at Wikoff Color Corporation will create one or more formulations to meet the performance and cost targets using the alternative pigment(s). Formulations that appear to meet the cost and performance requirements will be developed at larger scale to allow for testing the newly formulated ink in the Lewiston printing process.

6. Test the new ink formulations for iPCBs (Eurofins) EPA Method 1668 will be used to test the new formulation(s) for PCBs. Multiple tests (at least duplicate) of each new formulation will be submitted for testing to capture variability.

7. Compare the initial and revised formulations for cost, performance and PCB content

Based on the results of the performance trial, the cost estimates from Wikoff, and PCB test results a document will be prepared comparing these parameters for the current and proposed revised ink formulation.

8. Report results and proposed next steps for future work

The results of this pilot project will be summarized in a report and next steps will be recommended. Depending on the results, another pilot project focusing on alternative ink formulations for colors other than yellow may be proposed.

Timeline

Phase 1 of this project will begin upon approval of this proposal and will be completed by 30 June 2023. Additional work may be proposed pending results but funding of the additional work is outside of the scope of this proposal.

Budget

ChemFORWARD/HBN requires 50% payment at the start of the project to cover costs for testing and raw materials, and the remainder upon completion of tasks. The cost of this project includes the following:

- 1. Project management
- 2. PCB testing: Testing ink samples for PCBs using EPA Method 1668. The cost estimate is NOT TO EXCEED and includes shipping and/or other transportation costs. Testing will be done on at least two samples of each ink formulation. Testing will be performed on:
 - 1. Current ink formulations for 4 colors used by Lewiston (at least duplicate)
 - 2. Four samples from inks currently used by Northwest Offset Printing (in duplicate)
 - 3. Candidate yellow formulations designed by Wikoff (number tbd).
- 3. Research
 - **1.** Evaluation and specification of performance requirements and cost targets including collection of documentation from Lewiston
 - 2. Research and recommendations for alternative pigments (low or no-PCB containing)
 - 3. Help with evaluating PCB and performance test results
 - 4. Work with Wikoff to characterize the pigment(s) to optimize formulations
- 4. Development
 - 1. Procurement of pigments to meet yellow ink performance requirements using no/ultra-low PCB candidates
 - 2. Formulation of yellow inks with the alternative pigment(s) to meet performance and cost targets. Raw material costs included in the budget.
 - **3.** Testing of alternative ink performance in Lewiston presses including troubleshooting and formulation modifications to optimize performance.

- 5. Reporting
 - a. Periodic updates to the Task Force (virtual presentations)
 - **b.** PCB test results for all samples
 - **C.** Final report that compares the current and proposed revised ink formulations for:

14,400.00

31,350.00

12,000

4,000

50,000.00

2,000.00

1,200.00

114,950.00

11,495.00

126,445.00

160,445.00

- I. Presence of iPCBs
- ii. Cost
- iii. Performance
- **d.** Recommended next steps for the other ink colors (other than yellow).

Raw Material and Labor In-kind Hours Task Team Lead Rate Testing Costs Total (S) or Qty (CF) Contribution (Not to exceed) ChemFORWARD/Heine Project Management Lauren Heine 14,400 ŝ 200 72ŝ \$ 31,350.00 s 950 33 PCB Testing Eurofina Testing Lab ŝ ŝ ŝ Research & Development ChemFORWARD/Vincent Mark Vincent 200 12,000 S 60 5 4,000 Lauren Heine \$ 200 20 8 5 Wikoff, Chroma SC and Formulation R&D and printing labor (in-kind) \$ 200 170\$ 34,000.00 5 Lessisten. **Raw Materials** 50,000.00 (pigments, inks, and pilot material runs) Reporting \$ 200 10 2,000 Lauren Heine s \$ Mark Vincent \$ 200 б ŝ 1,200 ŝ \$ 34,000.00 \$ Subtotal 10% HBN Administrative Fee 5 Total cost of proposed work to SRRTTF \$ Total value with in-kind contribution ŝ

Table 1. Detailed Budget

About the Team

ChemFORWARD is a non-profit, value chain collaboration committed to providing trusted data on chemicals for use in consumer products. ChemFORWARD is a fiscally-sponsored project of the non-profit, Healthy Building Network (HBN). We believe that credible and robust hazard information and other relevant data on safety and sustainability underpins the pathway to safer products, and that increased use of such information will support proactive decision making and lead to safer products for all. This information is essential to enable a safe and circular economy. Our vision is to create the globally trusted source of cost-effective chemical hazard data for safer alternatives within a framework of safe and sustainable design. Lauren Heine will lead the project management for ChemFORWARD.

PROJECT TEAM

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Lewiston Printing/Revolve Print and Pack

Revolve Print and Pack is a family and employee-owned company born from a commitment to preserve, protect and champion our natural environment. An offshoot of a fourth-generation newspaper, Revolve's founders made the decision to not just report on issues of environmental significance, but to provide solutions that make a tangible difference. And like all successful ventures – it starts and ends with good, and passionate, people.

Northwest Offset Printing

Northwest Offset Printing is a local Washington company and a sister company to Inland Empire Paper. Northwest Offset Printing will contribute ink samples for testing for PCBs.

Wikoff Color Corporation:

Wikoff Color Corporation is a leading ink and coatings manufacturer for commercial printing applications. Wickoff is committed to producing high quality, tailor-made inks for the most challenging avenues of the printing.

Testing lab (Eurofins):

A testing lab in North America (Eurofins) will be commissioned to do the PCB testing. Individual tests are expected to be \sim \$950 each.

Mark Vincent, Ph.D., President of Chroma Specialty Chemicals

Dr. Mark Vincent will serve as research lead. Dr. Vincent is a highly experienced technical expert in color including dyes and pigments. He is a successful executive with a long track record of achieving corporate growth objectives through strategic plan development and implementation, providing diverse perspectives and positive leadership. Dr. Vincent has proven ability across multiple business functions including Operations, Sales, Marketing, R&D, Regulatory and Finance. He formerly served as the Group CEO and Technical Vice President of Dominion Colour Corporation in Toronto, Canada. Dr. Vincent earned his Ph.D. in Organic Color Chemistry at Cardiff University in Wales. His academic expertise and industry expertise will ensure research integrity and information produced that is practical and relevant to users. Contact: markvincent@chromasc.com; +1 (416) 702-9984; Toronto Canada; https://chromaspecialtychemicals.com/contact-us/

Grace Manarang-Pena, Vice President - Regulatory and Marketing

Since joining the pigment industry 25 years ago, Grace has become an expert in the global regulatory compliance of pigments as well as the development of specialty colored pigments. She spent over half her career in R&D before transitioning to the Global Regulatory, ISO and EHS Manager and Certified Toxic Reduction Planner at Dominion Colour Corporation in Toronto. Grace has been involved with Chemical Management Plan (CMP) activities in Canada, particularly with Azo substance grouping. With various associations in North America and Europe, she has participated in several Government interfaces and conferences, namely Chemcon. In 2014, Grace presented the process and lessons learned from the EU REACH Authorization Process, being the first to successfully apply for Authorization within the REACH

remit. Grace continues to monitor the global regulatory landscape for pigments and dyes. She works closely with manufacturers, customers, and associations to communicate the implications of chemical management programs (such as REACH) and GHS compliance. Contact: gracemanarang-pena@chromasc.com; +1 (289) 830 4232; Toronto Canada; https://chromaspecialtychemicals.com/contact-us/

ChemFORWARD/HBN

Lauren Heine, Ph.D., ChemFORWARD

Dr. Heine will serve as primary project manager and principal investigator for ChemFORWARD. She applies green chemistry, green engineering, alternatives assessment and multi-stakeholder collaboration to develop tools that result in safer and more sustainable chemical products and processes. Her work with Chem*FORWARD* builds on prior experience developing *GreenScreen®* for Safer Chemicals, a pioneering method for chemical hazard assessment to enable informed substitution; and *CleanGredients*TM, a web-based information platform for identifying greener chemicals for use in cleaning products; both tools were designed to scale access to information needed to develop materials and products that are safe and circular. Lauren worked closely with the US EPA Safer Choice Program to facilitate development of ingredient and hazard criteria for the Safer Choice Program.

For the SRRTTF, Dr. Heine worked in her capacity at Northwest Green Chemistry to provide several reports outlining potential green chemistry -- "beginning of product life" -- options to address the inadvertent PCB issue. For the OECD, Lauren drafted *Policy Principles for Sustainable Materials Management* and *Considerations and Criteria for Sustainable Plastics from a Chemicals Perspective*. She served on the California Green Ribbon Science Panel and co-chairs the Apple Green Chemistry Advisory Board. Lauren advised the technical development of the Interstate Chemicals Clearinghouse Alternatives Assessment Guide. She began her career as a Fellow with the American Association for the Advancement of Science in the Green Chemistry Program at the US Environmental Protection Agency. Lauren earned her doctorate in Civil and Environmental Engineering from Duke University.

Contact: lauren@chemforward.org; Mobile: 360.220.2069. Spokane, Washington

Appendix B. Names, Congener Numbers, and CAS Registry Numbers for Native and Labeled Chlorinated Biphenyl (CB) Congeners Determined by Isotope Dilution and Internal Standard HRGC/HRMS¹

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|---|---|------------------------|
| 2-MoCB | 1 | 2051-60-7 | ¹³ C ₁₂ -2-MoCB ² | 1L | 234432-85-0 |
| 3-MoCB | 2 | 2051-61-8 | | | |
| 4-MoCB | 3 | 2051-62-9 | ¹³ C ₁₂ -4-MoCB ² | 3L | 208263-77-8 |
| 2,2'-DiCB | 4 | 13029-08-8 | ¹³ C ₁₂ -2,2'-DiCB ² | 4L | 234432-86-1 |
| 2,3-DiCB | 5 | 16605-91-7 | | | |
| 2,3'-DiCB | 6 | 25569-80-6 | | | |
| 2,4-DiCB | 7 | 33284-50-3 | | | |
| 2,4-DiCB | 7 | 33284-50-3 | | | |
| 2,4'-DiCB ³ | 8 | 34883-43-7 | | | |
| 2,5-DiCB | 9 | 34883-39-1 | ¹³ C ₁₂ -2,5-DiCB ⁴ | 9L | 250694-89-4 |
| 2,6-DiCB | 10 | 33146-45-1 | | | |
| 3,3'-DiCB | 11 | 2050-67-1 | | | |

¹ The tables in Appendix B are excerpted from US EPA. April 2010. Method 1668C Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (Table 1) <u>https://www.epa.gov/sites/default/files/2015-09/documents/method_1668c_2010.pdf</u>

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|---|---|------------------------|
| 3,4-DiCB | 12 | 2974-92-7 | | | |
| 3,4'-DiCB | 13 | 2974-90-5 | | | |
| 3,5-DiCB | 14 | 34883-41-5 | | | |
| 4,4'-DiCB | 15 | 2050-68-2 | ¹³ C ₁₂ -4,4'-DiCB ² | 15L | 208263-67-6 |
| 2,2',3-TrCB | 16 | 38444-78-9 | | | |
| 2,2',4-TrCB | 17 | 37680-66-3 | | | |
| 2,2',5-TrCB ³ | 18 | 37680-65-2 | | | |
| 2,2',6-TrCB | 19 | 38444-73-4 | ¹³ C ₁₂ -2,2',6-TrCB ² | 19L | 234432-87-2 |
| 2,3,3'-TrCB | 20 | 38444-84-7 | | | |
| 2,3,4-TrCB | 21 | 55702-46-0 | | | |
| 2,3,4'-TrCB | 22 | 38444-85-8 | | | |
| 2,3,5-TrCB | 23 | 55720-44-0 | | | |
| 2,3,6-TrCB | 24 | 55702-45-9 | | | |
| 2,3',4-TrCB | 25 | 55712-37-3 | | | |
| 2,3',5-TrCB | 26 | 38444-81-4 | | | |
| 2,3',6-TrCB | 27 | 38444-76-7 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|--|---|------------------------|
| 2,4,4'-TrCB ³ | 28 | 7012-37-5 | ¹³ C ₁₂ -2,4,4'-TriCB ⁵ | 28L | 208263-76-7 |
| 2,4,5-TrCB | 29 | 15862-07-4 | | | |
| 2,4,6-TrCB | 30 | 35693-92-6 | | | |
| 2,4',5-TrCB | 31 | 16606-02-3 | | | |
| 2,4',6-TrCB | 32 | 38444-77-8 | | | |
| 2',3,4-TrCB | 33 | 38444-86-9 | | | |
| 2',3,5-TrCB | 34 | 37680-68-5 | | | |
| 3,3',4-TrCB | 35 | 37680-69-6 | | | |
| 3,3',5-TrCB | 36 | 38444-87-0 | | | |
| 3,4,4'-TrCB | 37 | 38444-90-5 | ¹³ C ₁₂ -3,4,4'-TrCB ² | 37L | 208263-79-0 |
| 3,4,5-TrCB | 38 | 53555-66-1 | | | |
| 3,4',5-TrCB | 39 | 38444-88-1 | | | |
| 2,2',3,3'-TeCB | 40 | 38444-93-8 | | | |
| 2,2',3,4-TeCB | 41 | 52663-59-9 | | | |
| 2,2',3,4'-TeCB | 42 | 36559-22-5 | | | |
| 2,2',3,5-TeCB | 43 | 70362-46-8 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|--|---|------------------------|
| 2,2',3,5'-TeCB ³ | 44 | 41464-39-5 | | | |
| 2,2',3,6-TeCB | 45 | 70362-45-7 | | | |
| 2,2',3,6'-TeCB | 46 | 41464-47-5 | | | |
| 2,2',4,4'-TeCB | 47 | 2437-79-8 | | | |
| 2,2',4,5-TeCB | 48 | 70362-47-9 | | | |
| 2,2',4,5'-TeCB | 49 | 41464-40-8 | | | |
| 2,2',4,6-TeCB | 50 | 62796-65-0 | | | |
| 2,2',4,6'-TeCB | 51 | 68194-04-7 | | | |
| 2,2',5,5'-TeCB ³ | 52 | 35693-99-3 | ¹³ C ₁₂ -2,2',5,5'-TeCB ⁴ | 52L | 208263-80-3 |
| 2,2',5,6'-TeCB | 53 | 41464-41-9 | | | |
| 2,2',6,6'-TeCB | 54 | 15968-05-5 | ¹³ C ₁₂ -2,2',6,6'-TeCB ² | 54L | 234432-88-3 |
| 2,3,3',4'-TeCB | 55 | 74338-24-2 | | | |
| 2,3,3',4'-TeCB | 56 | 41464-43-1 | | | |
| 2,3,3',5-TeCB | 57 | 70424-67-8 | | | |
| 2,3,3',5'-TeCB | 58 | 41464-49-7 | | | |
| 2,3,3',6-TeCB | 59 | 74472-33-6 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|---------------------|---|------------------------|
| 2,3,4,4'-TeCB | 60 | 33025-41-1 | | | |
| 2,3,4,5-TeCB | 61 | 33284-53-6 | | | |
| 2,3,4,6-TeCB | 62 | 54230-22-7 | | | |
| 2,3,4',5-TeCB | 63 | 74472-34-7 | | | |
| 2,3,4',6-TeCB | 64 | 52663-58-8 | | | |
| 2,3,5,6-TeCB | 65 | 33284-54-7 | | | |
| 2,3',4,4'-TeCB ³ | 66 | 32598-10-0 | | | |
| 2,3',4,5-TeCB | 67 | 73575-53-8 | | | |
| 2,3',4,5'-TeCB | 68 | 73575-52-7 | | | |
| 2,3',4,6-TeCB | 69 | 60233-24-1 | | | |
| 2,3',4',5-TeCB | 70 | 32598-11-1 | | | |
| 2,3',4',6-TeCB | 71 | 41464-46-4 | | | |
| 2,3',5,5'-TeCB | 72 | 41464-42-0 | | | |
| 2,3',5',6-TeCB | 73 | 74338-23-1 | | | |
| 2,4,4',5-TeCB | 74 | 32690-93-0 | | | |
| 2,4,4',6-TeCB | 75 | 32598-12-2 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|--|---|------------------------|
| 2',3,4,5-TeCB | 76 | 70362-48-0 | | | |
| 3,3',4,4'-TeCB ^{3,6} | 77 | 32598-13-3 | ¹³ C ₁₂ -3,3',4,4'-TeCB ^{2,7} | 77L | 105600-23-5 |
| 3,3',4,5-TeCB | 78 | 70362-49-1 | | | |
| 3,3',4,5'-TeCB | 79 | 41464-48-6 | | | |
| 3,3',5,5'-TeCB | 80 | 33284-52-5 | | | |
| 3,4,4',5-TeCB6 | 81 | 70362-50-4 | ¹³ C ₁₂ -3,4,4',5-TeCB ⁷ | 81L | 208461-24-9 |
| 2,2',3,3',4-PeCB | 82 | 52663-62-4 | | | |
| 2,2',3,3',5-PeCB | 83 | 60145-20-2 | | | |
| 2,2',3,3',6-PeCB | 84 | 52663-60-2 | | | |
| 2,2',3,4,4'-PeCB | 85 | 65510-45-4 | | | |
| 2,2',3,4,5-PeCB | 86 | 55312-69-1 | | | |
| 2,2',3,4,5'-PeCB | 87 | 38380-02-8 | | | |
| 2,2',3,4,6-PeCB | 88 | 55215-17-3 | | | |
| 2,2',3,4,6'-PeCB | 89 | 73575-57-2 | | | |
| 2,2',3,4',5-PeCB | 90 | 68194-07-0 | | | |
| 2,2',3,4',6-PeCB | 91 | 68194-05-8 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|--|---|------------------------|
| 2,2',3,5,5'-PeCB | 92 | 52663-61-3 | | | |
| 2,2',3,5,6-PeCB | 93 | 73575-56-1 | | | |
| 2,2',3,5,6'-PeCB | 94 | 73575-55-0 | | | |
| 2,2',3,5',6-PeCB | 95 | 38379-99-6 | | | |
| 2,2',3,6,6'-PeCB | 96 | 73575-54-9 | | | |
| 2,2',3',4,5-PeCB | 97 | 41464-51-1 | | | |
| 2,2',3',4,6-PeCB | 98 | 60233-25-2 | | | |
| 2,2',4,4',5-PeCB | 99 | 38380-01-7 | | | |
| 2,2',4,4',6-PeCB | 100 | 39485-83-1 | ¹³ C ₁₂ -2,2',4,5,5'-PeCB ⁴ | 101L | 104130-39-4 |
| 2,2',4,5,5'-PeCB ³ | 101 | 37680-73-2 | | | |
| 2,2',4,5,6'-PeCB | 102 | 68194-06-9 | | | |
| 2,2',4,5,'6-PeCB | 103 | 60145-21-3 | | | |
| 2,2',4,6,6'-PeCB | 104 | 56558-16-8 | ¹³ C ₁₂ -2,2',4,6,6'-PeCB ² | 104L | 234432-89-4 |
| 2,3,3',4,4'-PeCB ^{3,6} | 105 | 32598-14-4 | ¹³ C ₁₂ -2,3,3',4,4'-PeCB ⁷ | 105L | 208263-62-1 |
| 2,3,3',4,5-PeCB | 106 | 70424-69-0 | | | |
| 2,3,3',4',5-PeCB | 107 | 70424-68-9 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|--|---|------------------------|
| 2,3,3',4,5'-PeCB | 108 | 70362-41-3 | | | |
| 2,3,3',4,6-PeCB | 109 | 74472-35-8 | | | |
| 2,3,3',4',6-PeCB | 110 | 38380-03-9 | | | |
| 2,3,3',5,5'-PeCB | 111 | 39635-32-0 | ¹³ C ₁₂ -2,3,3',5,5'-PeCB ⁵ | 111 L | 235416-29-2 |
| 2,3,3',5,6-PeCB | 112 | 74472-36-9 | | | |
| 2,3,3',5',6-PeCB | 113 | 68194-10-5 | | | |
| 2,3,4,4',5-PeCB6 | 114 | 74472-37-0 | ¹³ C ₁₂ -2,3,4,4',5-PeCB ⁷ | 114 L | 208263-63-2 |
| 2,3,4,4',6-PeCB | 115 | 74472-38-1 | | | |
| 2,3,4,5,6-PeCB | 116 | 18259-05-7 | | | |
| 2,3,4',5,6-PeCB | 117 | 68194-11-6 | | | |
| 2,3',4,4',5-PeCB ^{3,6} | 118 | 31508-00-6 | ¹³ C ₁₂ -2',3,4,4',5-PeCB ⁷ | 123L | 208263-64-3 |
| 2,3',4,4',6-PeCB | 119 | 56558-17-9 | | | |
| 2,3',4,5,5'-PeCB | 120 | 68194-12-7 | | | |
| 2,3',4,5,'6-PeCB | 121 | 56558-18-0 | | | |
| 2',3,3',4,5-PeCB | 122 | 76842-07-4 | | | |
| 2',3,4,4',5-PeCB ⁶ | 123 | 65510-44-3 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|---|---|------------------------|
| 2',3,4,5,5'-PeCB | 124 | 70424-70-3 | | | |
| 2',3,4,5,6'-PeCB | 125 | 74472-39-2 | | | |
| 3,3',4,4',5-PeCB ^{3,6} | 126 | 57465-28-8 | ¹³ C ₁₂ -3,3',4,4',5-PeCB ^{2,7} | 126L | 208263-65-4 |
| 3,3',4,5,5'-PeCB | 127 | 39635-33-1 | | | |
| 2,2',3,3',4,4'-HxCB ³ | 128 | 38380-07-3 | | | |
| 2,2',3,3',4,5-HxCB | 129 | 55215-18-4 | | | |
| 2,2',3,3',4,5'-HxCB | 130 | 52663-66-8 | | | |
| 2,2',3,3',4,6-HxCB | 131 | 61798-70-7 | | | |
| 2,2',3,3',4,6'-HxCB | 132 | 38380-05-1 | | | |
| 2,2',3,3',5,5'-HxCB | 133 | 35694-04-3 | | | |
| 2,2',3,3',5,6-HxCB | 134 | 52704-70-8 | | | |
| 2,2',3,3',5,6'-HxCB | 135 | 52744-13-5 | | | |
| 2,2',3,3',6,6'-HxCB | 136 | 38411-22-2 | | | |
| 2,2',3,4,4',5-HxCB | 137 | 35694-06-5 | | | |
| 2,2',3,4,4',5'-HxCB ³ | 138 | 35065-28-2 | ¹³ C ₁₂ -2,2',3,4,4',5'-HxCB ⁴ | 138L | 208263-66-5 |
| 2,2',3,4,4',6-HxCB | 139 | 56030-56-9 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|---|---|------------------------|
| 2,2',3,4,4',6'-HxCB | 140 | 59291-64-4 | | | |
| 2,2',3,4,5,5'-HxCB | 141 | 52712-04-6 | | | |
| 2,2',3,4,5,6-HxCB | 142 | 41411-61-4 | | | |
| 2,2',3,4,5,6'-HxCB | 143 | 68194-15-0 | | | |
| 2,2',3,4,5',6-HxCB | 144 | 68194-14-9 | | | |
| 2,2',3,4,6,6'-HxCB | 145 | 74472-40-5 | | | |
| 2,2',3,4',5,5'-HxCB | 146 | 51908-16-8 | | | |
| 2,2',3,4',5,6-HxCB | 147 | 68194-13-8 | | | |
| 2,2',3,4',5,6'-HxCB | 148 | 74472-41-6 | | | |
| 2,2',3,4',5',6-HxCB | 149 | 38380-04-0 | | | |
| 2,2',3,4',6,6'-HxCB | 150 | 68194-08-1 | | | |
| 2,2',3,5,5',6-HxCB | 151 | 52663-63-5 | | | |
| 2,2',3,5,6,6'-HxCB | 152 | 68194-09-2 | | | |
| 2,2',4,4',5,5'-HxCB ³ | 153 | 35065-27-1 | | | |
| 2,2',4,4',5',6-HxCB | 154 | 60145-22-4 | | | |
| 2,2',4,4',6,6'-HxCB | 155 | 33979-03-2 | ¹³ C ₁₂ -2,2',4,4',6,6'-HxCB ² | 155L | 234432-90-7 |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|---|---|------------------------|
| 2,3,3',4,4',5-HxCB ⁶ | 156 | 38380-08-4 | ¹³ C ₁₂ -2,3,3',4,4',5-HxCB ⁷ | 156L | 208263-68-7 |
| 2,3,3',4,4',5'-HxCB6 | 157 | 69782-90-7 | ¹³ C ₁₂ -2,3,3',4,4',5'-HxCB ⁷ | 157L | 235416-30-5 |
| 2,3,3',4,4',6-HxCB | 158 | 74472-42-7 | | | |
| 2,3,3',4,5,5'-HxCB | 159 | 39635-35-3 | | | |
| 2,3,3',4,5,6-HxCB | 160 | 41411-62-5 | | | |
| 2,3,3',4,5',6-HxCB | 161 | 74472-43-8 | | | |
| 2,3,3',4',5,5'-HxCB | 162 | 39635-34-2 | | | |
| 2,3,3',4',5,6-HxCB | 163 | 74472-44-9 | | | |
| 2,3,3',4',5',6-HxCB | 164 | 74472-45-0 | | | |
| 2,3,3',5,5',6-HxCB | 165 | 74472-46-1 | | | |
| 2,3,4,4',5,6-HxCB | 166 | 41411-63-6 | | | |
| 2,3',4,4',5,5'-HxCB ⁶ | 167 | 52663-72-6 | ¹³ C ₁₂ -2,3',4,4',5,5'-HxCB ⁷ | 167L | 208263-69-8 |
| 2,3',4,4',5',6-HxCB | 168 | 59291-65-5 | | | |
| 3,3',4,4',5,5'-HxCB ^{3,6} | 169 | 32774-16-6 | ¹³ C ₁₂ -3,3',4,4',5,5'-HxCB ^{2,7} | 169L | 208263-70-1 |
| 2,2',3,3',4,4',5-HpCB ³ | 170 | 35065-30-6 | ¹³ C ₁₂ -2,2',3,3',4,4',5-HpCB | 170L | 160901-80-4 |
| 2,2'3,3',4,4',6-HpCB | 171 | 52663-71-5 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|--------------------------------------|--------------------|---------------------------|--|---|------------------------|
| 2,2',3,3',4,5,5'-HpCB | 172 | 52663-74-8 | | | |
| 2,2',3,3',4,5,6-HpCB | 173 | 68194-16-1 | | | |
| 2,2',3,3',4,5,6'-HpCB | 174 | 38411-25-5 | | | |
| 2,2',3,3',4,5',6-HpCB | 175 | 40186-70-7 | | | |
| 2,2',3,3',4,6,6'-HpCB | 176 | 52663-65-7 | | | |
| 2,2',3,3',4',5,6-HpCB | 177 | 52663-70-4 | | | |
| 2,2',3,3',5,5',6-HpCB | 178 | 52663-67-9 | ¹³ C ₁₂ -2,2',3,3',5,5',6-HpCB | 178L | 232919-67-4 |
| 2,2',3,3',5,6,6'-HpCB | 179 | 52663-64-6 | | | |
| 2,2',3,4,4',5,5'-HpCB ³ | 180 | 35065-29-3 | ¹³ C ₁₂ -2,2',3,4,4',5,5'-HpCB | 180L | 160901-82-6 |
| 2,2',3,4,4',5,6-HpCB | 181 | 74472-47-2 | | | |
| 2,2',3,4,4',5,6'-HpCB | 182 | 60145-23-5 | | | |
| 2,2',3,4,4',5',6-HpCB | 183 | 52663-69-1 | | | |
| 2,2',3,4,4',6,6'-HpCB | 184 | 74472-48-3 | | | |
| 2,2',3,4,5,5',6-HpCB | 185 | 52712-05-7 | | | |
| 2,2',3,4,5,6,6'-HpCB | 186 | 74472-49-4 | | | |
| 2,2',3,4',5,5',6-HpCB ³ | 187 | 52663-68-0 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|------------------------------------|--------------------|---------------------------|--|---|------------------------|
| 2,2',3,4',5,6,6'-HpCB | 188 | 74487-85-7 | ¹³ C ₁₂ -2,2',3,4',5,6,6'-HpCB | 188L | 234432-91-8 |
| 2,3,3',4,4',5,5'-HpCB ⁶ | 189 | 39635-31-9 | ¹³ C ₁₂ -2,3,3',4,4',5,5'-HpCB | 189L | 208263-73-4 |
| 2,3,3',4,4',5,6-HpCB | 190 | 41411-64-7 | | | |
| 2,3,3',4,4',5',6-HpCB | 191 | 74472-50-7 | | | |
| 2,3,3',4,5,5',6-HpCB | 192 | 74472-51-8 | | | |
| 2,3,3',4',5,5',6-HpCB | 193 | 69782-91-8 | | | |
| 2,2',3,3',4,4',5,5'-OcC B | 194 | 35694-08-7 | | | |
| 2,2',3,3',4,4',5,6-OcCB 3 | 195 | 52663-78-2 | | | |
| 2,2',3,3',4,4',5,6'-OcC B | 196 | 42740-50-1 | | | |
| 2,2',3,3',4,4',6,6'-OcC B | 197 | 33091-17-7 | | | |
| 2,2',3,3',4,5,5',6-OcCB | 198 | 68194-17-2 | | | |
| 2,2',3,3',4,5,5',6'-OcC B | 199 | 52663-75-9 | | | |
| 2,2',3,3',4,5,6,6'-OcCB | 200 | 52663-73-7 | | | |

| CB congener name ¹ | Congener number | CAS Registry number | Labeled analog name | Labeled analog congener number | CAS Registry number |
|---------------------------------|--------------------|---------------------------|--|---|------------------------|
| 2,2',3,3',4,5',6,6'-OcC B | 201 | 40186-71-8 | | | |
| 2,2',3,3',5,5',6,6'-OcC B | 202 | 2136-99-4 | ¹³ C ₁₂ -2,2',3,3',5,5',6,6'-Oc CB ² | 202L | 105600-26-8 |
| 2,2',3,4,4',5,5',6-OcCB | 203 | 52663-76-0 | | | |
| 2,2',3,4,4',5,6,6'-OcCB | 204 | 74472-52-9 | | | |
| 2,3,3',4,4',5,5',6-OcCB | 205 | 74472-53-0 | ¹³ C ₁₂ -2,3,3',4,4',5,5',6-Oc CB ² | 205L | 234446-64-1 |
| 2,2',3,3',4,4',5,5',6-No CB3 | 206 | 40186-72-9 | ¹³ C ₁₂ -2,2',3,3',4,4',5,5',6- NoCB ² | 206L | 208263-75-6 |
| 2,2',3,3',4,4',5,6,6'-No CB | 207 | 52663-79-3 | | | |
| 2,2',3,3',4,5,5',6,6'-No CB | 208 | 52663-77-1 | ¹³ C ₁₂ -2,2',3,3',4,5,5',6,6'- NoCB ² | 208L | 234432-92-9 |
| DeCB ³ | 209 | 2051-24-3 | ¹³ C ₁₂ -DeCB ² | 209L | 105600-27-9 |

1. Abbreviations for chlorination levels

| МоСВ | monochlorobiphenyl | OcCB | octachlorobiphenyl |
|------|---------------------|------|---------------------|
| НхСВ | hexachlorobiphenyl | ТеСВ | tetrachlorobiphenyl |
| DiCB | dichlorobiphenyl | NoCB | nonachlorobiphenyl |
| НрСВ | heptachlorobiphenyl | PeCB | pentachlorobiphenyl |

- 2. Labeled level of chlorination (LOC) window-defining congener
- 3. National Oceanic and Atmospheric Administration (NOAA) congener of interest
- 4. Labeled injection internal standard
- 5. Labeled clean-up standard
- 6. World Health Organization (WHO) toxic congener
- 7. Labeled analog of WHO toxic congener

Table A-2. QC Acceptance Criteria for VER, IPR, OPR, and Labeled Compounds in Samples¹

| Congener Name | Congener no | IPR RSD (50%) | IPR Mean Recovery (%) | OPR Recovery (%) | Labeled Compound Recovery in Samples (%) |
|------------------|----------------|---------------------|-----------------------------|------------------------|--|
| 2-MoCB | 1 | 25 | 70 - 130 | 60 - 135 | |
| 4-MoCB | 3 | 25 | 70 - 130 | 60 - 135 | |
| 2,2'-DiCB | 4 | 25 | 70 - 130 | 60 - 135 | |
| 4,4'-DiCB | 15 | 25 | 70 - 130 | 60 - 135 | |
| 2,2'6-TrCB | 19 | 25 | 70 - 130 | 60 - 135 | |
| 3,4,4'-TrCB | 37 | 25 | 70 - 130 | 60 - 135 | |
| 2,2'6,6'TeCB | 54 | 25 | 70 - 130 | 60 - 135 | NA |
| 3,3',4,4'-TeCB | 77 | 25 | 70 - 130 | 60 - 135 | |
| 3,4,4',5-TeCB | 81 | 25 | 70 - 130 | 60 - 135 | |
| 2,2',4,6,6'-PeCB | 104 | 25 | 70 - 130 | 60 - 135 | |
| 2,3,3',4,4'-PeCB | 105 | 25 | 70 - 130 | 60 - 135 | |
| 2,3,4,4',5-PeCB | 114 | 25 | 70 - 130 | 60 - 135 | |
| 2,3',4,4',5-PeCB | 118 | 25 | 70 - 130 | 60 - 135 | |

| Congener Name | Congener no | IPR RSD (50%) | IPR Mean Recovery (%) | OPR Recovery (%) | Labeled Compound Recovery in Samples (%) |
|--|----------------|---------------------|-----------------------------|------------------------|--|
| 2',3,4,4',5-PeCB | 123 | 25 | 70 - 130 | 60 - 135 | |
| 3,3',4,4',5-PeCB | 126 | 25 | 70 - 130 | 60 - 135 | |
| 2,2',4,4',6,6'-HxCB | 155 | 25 | 70 - 130 | 60 - 135 | |
| 2,3,3',4,4',5-HxCB ⁶ | 156 | 25 | 70 - 130 | 60 - 135 | |
| 2,3,3',4,4',5'-HxCB ⁶ | 157 | 25 | 70 - 130 | 60 - 135 | |
| 2,3',4,4',5,5'-HxCB | 167 | 25 | 70 - 130 | 60 - 135 | |
| 3,3',4,4',5,5'-HxCB | 169 | 25 | 70 - 130 | 60 - 135 | |
| 2,2',3,4',5,6,6'-HpCB | 188 | 25 | 70 - 130 | 60 - 135 | |
| 2,3,3',4,4',5,5'-HpCB | 189 | 25 | 70 - 130 | 60 - 135 | |
| 2,2',3,3',5,5',6,6'-OcCB | 202 | 25 | 70 - 130 | 60 - 135 | |
| 2,3,3',4,4',5,5',6-OcCB | 205 | 25 | 70 - 130 | 60 - 135 | |
| 2,2',3,3',4,4',5,5',6-NoCB | 206 | 25 | 70 - 130 | 60 - 135 | |
| 2,2',3,3,'4,5,5',6,6'-NoCB | 208 | 25 | 70 - 130 | 60 - 135 | |
| DeCB | 209 | 25 | 70 - 130 | 60 - 135 | |
| ¹³ C ₁₂ -2-MoCB | 1L | 70 | 20 - 135 | 15 - 145 | 5 - 145 |
| ¹³ C ₁₂ -4-MoCB | 3L | 70 | 20 - 135 | 15 - 145 | 5 - 145 |
| ¹³ C ₁₂ -2,2'-DiCB | 4L | 70 | 20 - 135 | 15 - 145 | 5 - 145 |
| ¹³ C ₁₂ -4,4'-DiCB | 15L | 70 | 20 - 135 | 15 - 145 | 5 - 145 |
| ¹³ C ₁₂ -2,2',6-TrCB | 19L | 70 | 20 - 135 | 15 - 145 | 5 - 145 |

| Congener Name | Congener no | IPR RSD (50%) | IPR Mean Recovery (%) | OPR Recovery (%) | Labeled Compound Recovery in Samples (%) |
|---|----------------|---------------------|-----------------------------|------------------------|--|
| ¹³ C ₁₂ -3,4,4'-TrCB | 37L | 70 | 20 - 135 | 15 - 145 | 5 - 145 |
| ¹³ C ₁₂ -2,2',6,6'-TeCB | 54L | 70 | 20 - 135 | 15 - 145 | 5 - 145 |
| ¹³ C ₁₂ -3,3',4,4'-TeCB | 77L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -3,4,4',5-TeCB | 81L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2,2',4,6,6'-PeCB | 104L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2,3,3',4,4'-PeCB | 105L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2,3,4,4',5-PeCB | 114L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2,3',4,4',5-PeCB | 118L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2',3,4,4',5-PeCB | 123L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -3,3',4,4',5-PeCB | 126L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2,2',4,4',6,6'-HxCB | 155L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2,3,3',4,4',5 -HxCB | 156L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2,3,3',4,4',5'-HxCB | 157L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2,3',4,4',5,5'-HxCB | 167L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -3,3',4,4',5,5'-HxCB | 169L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2,2',3,4',5,6,6'-HpCB | 188L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |
| ¹³ C ₁₂ -2',3,3',4,4',5,5'-HpCB | 189L | 50 | 45 - 135 | 40 - 145 | 10 - 145 |

| Congener Name | Congener no | IPR RSD (50%) | IPR Mean Recovery (%) | OPR Recovery (%) | Labeled Compound Recovery in Samples (%) | | |
|--|----------------|---------------------|-----------------------------|------------------------|--|--|--|
| ¹³ C ₁₂ -2,2',3,3',5,5',6,6'-OcCB | 202L | 50 | 45 - 135 | 40 - 145 | 10 - 145 | | |
| ¹³ C ₁₂ -2,3,3',4,4',5,5',6-OcCB | 205L | 50 | 45 - 135 | 40 - 145 | 10 - 145 | | |
| ¹³ C ₁₂ -2,2',3,3',4,4',5,5',6-NoCB | 206L | 50 | 45 - 135 | 40 - 145 | 10 - 145 | | |
| ¹³ C ₁₂ -2,2',3,3',4,5,5',6,6'-NoCB | 208L | 50 | 45 - 135 | 40 - 145 | 10 - 145 | | |
| ¹³ C ₁₂ -2,2',3,3',4,4',5,5',6,6'-DeCB | 209L | 50 | 45 - 135 | 40 - 145 | 10 - 145 | | |
| Cleanup standards | | | | | | | |
| ¹³ C ₁₂ -2,4,4'-TrCB | 28L | 70 | 20 - 135 | 15 - 145 | 5 - 145 | | |
| ¹³ C ₁₂ -2,3,3',5,5'-PeCB | 111L | 50 | 45 - 135 | 40 - 145 | 10 - 145 | | |
| ¹³ C ₁₂ -2,2',3,3',5,5',6-HpCB | 178L | 50 | 45 - 135 | 40 - 145 | 10 - 145 | | |

Appendix C: Glossary & Abbreviations

Accreditation: A certification process for laboratories, designed to evaluate and document a lab's ability to perform analytical methods and produce acceptable data (Kammin, 2010). For Ecology, it is defined according to WAC 173-50-040: "Formal recognition by [Ecology] that an environmental laboratory is capable of producing accurate and defensible analytical data."

Accuracy: The degree to which a measured value agrees with the true value of the measured property. USEPA recommends that this term not be used, and that the terms *precision* and *bias* be used to convey the information associated with the term *accuracy* (USEPA, 2014).

Analyte: An element, ion, compound, or chemical moiety (pH, alkalinity) which is to be determined. The definition can be expanded to include organisms, e.g., fecal coliform, Klebsiella (Kammin, 2010).

Bias: Discrepancy between the expected value of an estimator and the population parameter being estimated (Gilbert, 1987; USEPA, 2014).

Blank: A synthetic sample, free of the analyte(s) of interest. For example, in water analysis, pure water is used for the blank. In chemical analysis, a blank is used to estimate the analytical response to all factors other than the analyte in the sample. In general, blanks are used to assess possible contamination or inadvertent introduction of analyte during various stages of the sampling and analytical process (USGS, 1998).

Calibration: The process of establishing the relationship between the response of a measurement system and the concentration of the parameter being measured (Ecology, 2004).

Check standard: A substance or reference material obtained from a source independent from the source of the calibration standard; used to assess bias for an analytical method. This is an obsolete term, and its use is highly discouraged. See Calibration Verification Standards, Lab Control Samples (LCS), Certified Reference Materials (CRM), and/or spiked blanks. These are all check standards but should be referred to by their actual designator, e.g., CRM, LCS (Kammin, 2010; Ecology, 2004).

Comparability: The degree to which different methods, data sets and/or decisions agree or can be represented as similar; a data quality indicator (USEPA, 2014; USEPA, 2020).

Completeness: The amount of valid data obtained from a project compared to the planned amount. Usually expressed as a percentage. A data quality indicator (USEPA, 2014; USEPA 2020).

Continuing Calibration Verification Standard (CCV): A quality control (QC) sample analyzed with samples to check for acceptable bias in the measurement system. The CCV is usually a midpoint calibration standard that is re-run at an established frequency during the course of an analytical run (Kammin, 2010).

Control chart: A graphical representation of quality control results demonstrating the performance of an aspect of a measurement system (Kammin, 2010; Ecology 2004).

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Control limits: Statistical warning and action limits calculated based on control charts. Warning limits are generally set at +/- 2 standard deviations from the mean, action limits at +/- 3 standard deviations from the mean (Kammin, 2010).

Data integrity: A qualitative DQI that evaluates the extent to which a data set contains data that is misrepresented, falsified, or deliberately misleading (Kammin, 2010).

Data quality indicators (DQI): Commonly used measures of acceptability for environmental data. The principal DQIs are precision, bias, representativeness, comparability, completeness, sensitivity, and integrity (USEPA, 2006).

Data quality objectives (DQO): Qualitative and quantitative statements derived from systematic planning processes that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions (USEPA, 2006).

Data set: A grouping of samples organized by date, time, analyte, etc. (Kammin, 2010).

Data validation: The process of determining that the data satisfy the requirements as defined by the data user (USEPA, 2020). There are various levels of data validation (USEPA, 2009).

Data verification: Examination of a data set for errors or omissions, and assessment of the Data Quality Indicators related to that data set for compliance with acceptance criteria (MQOs). Verification is a detailed quality review of a data set (Ecology, 2004).

Detection limit (limit of detection): The concentration or amount of an analyte which can be determined to a specified level of certainty to be greater than zero (Ecology, 2004).

Duplicate samples: Two samples taken from and representative of the same population, and carried through and steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variability of all method activities including sampling and analysis (USEPA, 2014).

Ecology: Washington State Department of Ecology

Field blank: A blank used to obtain information on contamination introduced during sample collection, storage, and transport (Ecology, 2004).

Initial Calibration Verification Standard (ICV): A QC sample prepared independently of calibration standards and analyzed along with the samples to check for acceptable bias in the measurement system. The ICV is analyzed prior to the analysis of any samples (Kammin, 2010).

Laboratory Control Sample (LCS)/LCS duplicate: A sample of known composition prepared using contaminant-free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is prepared and analyzed in the same batch of regular samples using the same sample preparation method, reagents, and analytical methods employed for regular samples. Monitors a lab's performance for bias and precision (USEPA, 2014).

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Matrix spike/Matrix spike duplicate: A QC sample prepared by adding a known amount of the target analyte(s) to an aliquot of a sample to check for bias and precision errors due to interference or matrix effects (Ecology, 2004).

Measurement Quality Objectives (MQOs): Performance or acceptance criteria for individual data quality indicators, usually including precision, bias, sensitivity, completeness, comparability, and representativeness (USEPA, 2006).

Measurement result: A value obtained by performing the procedure described in a method (Ecology, 2004).

Method: A formalized group of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, data analysis), systematically presented in the order in which they are to be executed (USEPA, 2001).

Method blank: A blank prepared to represent the sample matrix, prepared and analyzed with a batch of samples. A method blank will contain all reagents used in the preparation of a sample, and the same preparation process is used for the method blank and samples (Ecology, 2004; Kammin, 2010).

Method Detection Limit (MDL): The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results (USEPA, 2016). MDL is a measure of the capability of an analytical method of distinguished samples that do not contain a specific analyte from a sample that contains a low concentration of the analyte (USEPA, 2020).

Minimum level: Either the sample concentration equivalent to the lowest calibration point in a method or a multiple of the method detection limit (MDL), whichever is higher. For the purposes of NPDES compliance monitoring, EPA considers the following terms to be synonymous: "quantitation limit," "reporting limit," and "minimum level" (40 CFR 136).

Parameter: A specified characteristic of a population or sample. Also, an analyte or grouping of analytes. Benzene and nitrate + nitrite are all parameters (Kammin, 2010; Ecology, 2004).

Population: The hypothetical set of all possible observations of the type being investigated (Ecology, 2004).

Precision: The extent of random variability among replicate measurements of the same property; a data quality indicator (USGS, 1998).

Quality assurance (QA): A set of activities designed to establish and document the reliability and usability of measurement data (Kammin, 2010).

Quality Assurance Project Plan (QAPP): A document that describes the objectives of a project, and the processes and activities necessary to develop data that will support those objectives (Kammin, 2010; Ecology, 2004).

Quality control (QC): The routine application of measurement and statistical procedures to assess the accuracy of measurement data (Ecology, 2004).

Relative Percent Difference (RPD): RPD is commonly used to evaluate precision. The following formula is used:

$$RPD = [Abs(a-b)/((a + b)/2)] * 100\%$$

where "Abs()" is absolute value and a and b are results for the two replicate samples. RPD can be used only with 2 values. Percent Relative Standard Deviation is (%RSD) is used if there are results for more than 2 replicate samples (Ecology, 2004).

Relative Standard Deviation (RSD): A statistic used to evaluate precision in environmental analysis. It is determined in the following manner:

$$RSD = (100\% * s)/x$$

where s is the sample standard deviation and x is the mean of results from more than two replicate samples (Kammin, 2010).

Replicate samples: Two or more samples taken from the environment at the same time and place, using the same protocols. Replicates are used to estimate the random variability of the material sampled (USGS, 1998).

Reporting level: Unless specified otherwise by a regulatory authority or in a discharge permit, results for analytes that meet the identification criteria (i.e., rules for determining qualitative presence/absence of an analyte) are reported down to the concentration of the minimum level established by the laboratory through calibration of the instrument. EPA considers the terms "reporting limit," "quantitation limit," and "minimum level" to be synonymous (40 CFR 136).

Representativeness: The degree to which a sample reflects the population from which it is taken; a data quality indicator (USGS, 1998).

Sample (field): A portion of a population (environmental entity) that is measured and assumed to represent the entire population (USGS, 1998).

Sample (statistical): A finite part or subset of a statistical population (USEPA, 1992).

Sensitivity: In general, denotes the rate at which the analytical response (e.g., absorbance, volume, meter reading) varies with the concentration of the parameter being determined. In a specialized sense, it has the same meaning as the detection limit (Ecology, 2004).

Spiked blank: A specified amount of reagent blank fortified with a known mass of the target analyte(s); usually used to assess the recovery efficiency of the method (USEPA, 2014).

Spiked sample: A sample prepared by adding a known mass of target analyte(s) to a specified amount of matrix sample for which an independent estimate of target analyte(s) concentration is available. Spiked samples can be used to determine the effect of the matrix on a method's recovery efficiency (USEPA, 2014).

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Split sample: A discrete sample subdivided into portions, usually duplicates (Kammin, 2010).

Standard Operating Procedure (SOP): A document which describes in detail a reproducible and repeatable organized activity (Kammin, 2010).

Surrogate: For environmental chemistry, a surrogate is a substance with properties similar to those of the target analyte(s). Surrogates are unlikely to be native to environmental samples. They are added to environmental samples for quality control purposes, to track extraction efficiency and/or measure analyte recovery. Deuterated organic compounds are examples of surrogates commonly used in organic compound analysis (Kammin, 2010).

Systematic planning: A step-wise process which develops a clear description of the goals and objectives of a project, and produces decisions on the type, quantity, and quality of data that will be needed to meet those goals and objectives. The DQO process is a specialized type of systematic planning (USEPA, 2006).

US EPA: United States Environmental Protection Agency

Appendix D: References for Quality Assurance Terms in Glossary and for Guidances for Preparation of a Quality Assurance Project Plan

- 40 CFR 136. Title 40 Code of Federal Regulations, Part 136: Guidelines Establishing Test Procedures for the Analysis of Pollutants. Available at: <u>https://www.ecfr.gov/cgi-bin/text-idx?SID=3cf9acace214b7af340ea8f6919a7c39&mc=tru</u> <u>e&node=pt40.25.136&rgn=div5</u> (accessed 26 Feb. 2020).
- Ecology, 2004. Guidance for the Preparation of Quality Assurance Project Plans for Environmental Studies. Washington State Department of Ecology, Olympia, WA. Available at: <u>https://fortress.wa.gov/ecy/publications/SummaryPages/0403030.html</u> (accessed 6 Mar. 2020).
- Gilbert, R.O., 1987. Statistical Methods for Environmental Pollution Monitoring. Van Nostrand Reinhold, New York, NY.
- Kammin, W., 2010. Definition developed or extensively edited by William Kammin, 2010. Washington State Department of Ecology, Olympia, WA.
- USEPA, 1992. Guidelines for exposure assessment. U.S. Environmental Protection Agency, Risk Assessment Forum, Washington, D.C. EPA/600/Z-92/001. Available at: https://www.epa.gov/sites/production/files/2014-11/documents/guidelines_exp_assess ment.pdf (accessed 26 Feb. 2020).
- USEPA, 2001. EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5. U.S. Environmental Protection Agency, Washington, DC. EPA/240/B-01/003. Available at: <u>https://www.epa.gov/quality/epa-qar-5-epa-requirements-quality-assurance-project-plan</u> <u>s</u> (accessed 26 Feb. 2020).
- USEPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process EPA QA/G-4. U.S. Environmental Protection Agency, Washington, DC. <u>Available at:</u> <u>https://www.epa.gov/sites/production/files/2015-06/documents/g4-final.pdf</u> (accessed 26 Feb. 2020).
- USEPA, 2009. Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use, OSWER No. 9200.1-85, EPA 540-R-08-005. U.S. Environmental Protection Agency, Washington, DC. Available at: <u>https://www.epa.gov/nscep</u>.
- USEPA, 2014. Compendium: Project Quality Assurance and Quality Control: Chapter 1. U.S. Environmental Protection Agency, Washington, DC. SW-846 Update V. Available at: <u>https://www.epa.gov/sites/production/files/2015-10/documents/chap1_1.pdf</u> (accessed 26 Feb. 2020).
- USEPA, 2016. Definition and Procedure for the Determination of the Method Detection Limit, Revision 2. EPA 821-R-16-006. U.S. Environmental Protection Agency, Washington, DC. Available at: <u>https://www.epa.gov/sites/production/files/2016-12/documents/mdl-procedure_rev2_1</u> <u>2-13-2016.pdf</u> (accessed 6 Mar. 2020).

- USEPA, 2020. Glossary: Environmental Sampling and Analytical Methods (ESAM) Program. U.S. Environmental Protection Agency, Washington, DC. Available at: https://www.epa.gov/esam/glossary (accessed 26 Feb. 2020).
- USGS, 1998. Principles and Practices for Quality Assurance and Quality Control. Open-File Report 98-636. U.S. Geological Survey, Reston, VA. Available at: https://pubs.usgs.gov/of/1998/ofr98-636/ (accessed 26 Feb. 2020).
- WAC 173-50-040. Title 173 Washington Administrative Code. Accreditation of Environmental Laboratories: Definitions. Available at: https://apps.leg.wa.gov/WAC/default.aspx?cite=173-50-040 (accessed 26 Feb. 2020).
- Washington Department of Ecology. July 2004 (Revised December 2016). Guidelines for Preparing Quality Assurance Project Plans for Environmental Studies. Publication No. 04-03-030 (Revision of Publication No. 01-03-003) LINK
- Washington Department of Ecology (WA DOE). March 2022. Standard Operating Procedure PTP002, Version 2.1. Product Testing Database for Data Entry and Data Entry Quality Assurance. Publication 22-03-208. LINK
- Washington Department of Ecology (WA DOE). May 2021. Product Testing Standard Operating Procedure PTP001, Version 2.1. Product Collection and Sample Processing. Publication 21-03-21. LINK

Washington Department of Ecology (WA DOE). Accessed 2023. Quality Assurance website. LINK

Washington Department of Ecology (WA DOE). June 2021. Quality Assurance Project Plan. Perand Polyfluoroalkyl Substances (PFAS) in Washington State Products. Product Testing Program. Publication 18-04-012 LINK