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State of Washington

PCBs in State Purchased Products - 2017

Addendum to Quality Assurance Project Plan: Product Testing Program, Version 1.0

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This is an addendum to the Quality Assurance Project Plan: Product Testing Program, Version 1.0 (publication no. 16-03-113). It is not a correction (errata) to an original plan.

Data for this study will be available on the Product Testing Database (PTDB) website at <http://ecyapeem/ptdbpublicreporting/>. Search Study: PCBs in State Purchased Products – 2017.

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HWTR: Hazardous Waste and Toxics Reduction Program
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2.0 Abstract

The Washington State Department of Ecology (Ecology) will conduct a study to evaluate the presence of polychlorinated biphenyls (PCBs) in five categories of state purchased products. Washington State law (RCW [39.26.280](#) and [39.26.290](#)) requires state agencies to limit the purchase of products containing PCBs. This study is being carried out to assist state agencies in identifying where PCBs may be present in a subgroup of state purchased products from state contracts in order to comply with the law. A final report summarizing findings will be published. All data will be entered into a publicly available database on Ecology's website at <http://ecyapeem/ptdbpublicreporting/>.

3.0 Background

In 2013, the Washington State Department of Ecology (Ecology) initiated a study to evaluate the presence of polychlorinated biphenyls (PCBs) in general consumer products (Ecology, 2013). Particular emphasis was placed on testing products known to contain PCBs as a contaminant formed during the manufacturing of dyes and pigments within the product or from the use of chlorinated compounds in the manufacturing process (NYAS, 2005). PCB-containing products identified by previous research include paints, newspapers, glossy magazines, cereal boxes, and yellow plastic bags (Hu, 2010; Rodenburg, 2012).

In the 2013 Ecology study, 68 products were separated into 74 samples and tested for four PCB congeners (PCB-11, PCB-206, PCB-208 and PCB-209) indicative of inadvertent PCB production during the manufacturing of pigments and dyes. PCB congeners is a term used for the family of 209 compounds similar in structure with different chlorination patterns of biphenyl (NYAS, 2005). The types of product samples analyzed included packaging, paper products, paints, paint colorants, and caulks. Results showed almost all paint and paint colorant samples contained one or more of the four PCB congeners at detectable levels (Ecology, 2014a). Packaging and paper products sampled also contained PCBs, particularly PCB-11. PCB-206 and -208 were not present in most of the products tested, while seven samples contained PCB-209.

In 2014, Ecology initiated a follow-up study to expand the types of products sampled and PCBs analyzed in the original study (Ecology, 2014b). For this study, 133 additional products were separated into 142 samples and tested for the full suite of 209 PCB congeners. Product samples included children's clothing, silk screening and clothing dyes, cosmetics, body care products, comic books, office supplies, food packaging, road paints, lawn and road care, and an adult uniform. Several of these samples were collected from state purchased products: food packaging, road paints, and an adult uniform.

The 74 samples from the 2013 study were re-analyzed to obtain results for the full suite of 209 PCB congeners and total PCBs. This data was combined with the 2014 study results of 142 samples tested for individual and total PCBs. The combined 216 sample results from 201 consumer products were published into one report (Ecology, 2016b). A total of 216 samples were analyzed from 201 consumer products. The results showed 89% of the samples (193 of

216 samples) selected for testing contained detectable total PCBs above the method reporting limit (MRL). Three samples contained total PCBs over 1,000 ppb (parts per billion): a child's yellow sidewalk chalk, a single-serving cereal packaging, and a yellow foam office product. These studies found PCB contamination in a variety of consumer products, including a subgroup of state purchased products.

4.0 Study Description

This study will focus on testing selected state purchased products for PCBs. This addendum to the Quality Assurance Project Plan (QAPP): Product Testing Program, Version 1.0 (Ecology, 2016a) describes project specific information and deviations from the overall product testing QAPP. Washington State law [Revised Code of Washington (RCW) [39.26.280](#)] requires the Department of Enterprise Services (DES) to establish a procurement preference for products and packaging that does not contain PCBs. The law also grants state agencies the authority to require manufacturers to provide test results documenting the level of PCBs in products and packaging (RCW [39.26.290](#)).

4.1 Study Goals

This study is being carried out with the following goals:

- Assist DES with determining which products on state contracts comply with the PCBs in state purchased products law (RCW [39.26.280](#)).
- Identify state purchased products that contain PCBs and that could be released into the environment.

4.2 Study Objectives

Study objectives include the following:

- Collect and test state purchased products (products that the state currently purchases under contract) for PCB congeners.

The selection criteria for products in this study include:

- State purchased products from the selected product categories below.
- Products suspected to contain PCBs (due to the manufacturing process).
- Products that are or could be released into the environment.

The product categories selected for this study include but are not limited to:

- A. **Fish Hatchery:** Sample state-purchased products, like fish feed and fish pharmaceuticals, that are introduced directly into the fish hatchery water. A previous study reported the presence of PCBs in fish hatchery feed in the State of Washington using a different analytical method to test for PCBs (Ecology, 2006).

- B. **Janitorial Supplies:** Sample state purchased products that contain dyes and pigments or use chlorinated compounds in the manufacturing process that can be discharged down the drain or disposed of in trash.
- C. **Lubricants:** Sample a variety of state purchased lubricants used in state vehicles and ferries that could leak onto the roads or into the watershed. A previous study reported the presence of PCBs in five samples of motor oil and vehicle lubricants (City of Spokane, 2015).
- D. **Medical & Hospital Supplies:** Sample state purchased products that contain dyes and pigments or use chlorinated compounds in the manufacturing process that are often used once and disposed of either into the landfill or by incineration.
- E. **Flooring Material:** Sample state purchased products, like carpeting, that contain dyes and pigments or use chlorinated compounds in the manufacturing process that are purchased in large volumes, used in state buildings, and disposed of into the landfill.

5.0 Organization and Schedule

5.4 Study schedule

Table 1. Proposed Schedule for Completing Product Collection and Laboratory Work, Data Reviews, Data Entry into Product Testing Database (PTDB), and Reports

Product Collection and Laboratory Work	Due Date	Lead Staff
Product collection completed	03/2017	Kari Trumbull/Chrissy Wiseman
Product logging in completed	03/2017	Chrissy Wiseman
XRF screening completed	03/2017	Chrissy Wiseman
Internal data QA completed	03/2017	Chrissy Wiseman
Laboratory analyses completed and received	06/2017	
Data Review	Due Date	Lead Staff
Lab data QA reviewed	07/2017	Kari Trumbull
Lab data loaded into PTDB	08/2017	Kari Trumbull
PTDB data QA review completed	09/2017	Kari Trumbull/Chrissy Wiseman
Final Report	Due Date	Lead Staff
Draft due to supervisor	10/2017	Kari Trumbull
Draft due to client/peer reviewer	10/2017	Kari Trumbull
Final report due to publications coordinator	11/2017	Kari Trumbull
Final report posted to the web	12/2017	

5.6 Budget and Funding

An estimated 60 samples will be sent to a contract lab and analyzed for the full suite of 209 PCB congeners. An estimated 10% of samples (6 of the 60 samples) is included in the budget to account for cryomilling. Some samples may need to be cryomilled, like flooring material, to obtain a homogenous sample before extraction and analysis.

Ecology's Manchester Environmental Laboratory (MEL) will perform the analysis of titanium on duplicate samples for an estimated 6 of the 60 samples sent to the lab for PCB analysis. These samples will be selected from samples that have detectable levels of titanium from XRF screening.

Table 2. Study Budget and Funding

Activity/Parameter	Number of Samples	QC Samples ⁺	Cost of Sample	Subtotal	Total
Product Collection	60	---	\$ 20	\$ 1,200	---
Product Collection Total:					\$ 1,200
Cryomilling	6	---	\$ 100	\$ 600	---
209 PCB Congeners [*]	60	10	\$ 725	\$ 50,750	---
Titanium	6	3	\$ 70	\$ 630	---
Laboratory Total:					\$ 51,980
Study Total:					\$ 53,180

⁺QC samples in this table include those that are not provided free of charge (matrix spikes, matrix spike duplicates, sample duplicates, and cryomill rinseates).

^{*}Price includes 25% MEL contract fee.

6.0 Quality Objectives

6.2 Measurement Quality Objectives

Measurement Quality Objectives (MQOs) for PCB and titanium laboratory analyses are shown in [Table 3](#). MQOs for this study are to obtain data of sufficient quality to determine concentrations of the 209 individual PCB congeners and calculate total PCB concentrations (sum of PCB congeners) in samples from state purchased products, and in a subset of duplicate samples for titanium analysis. MQOs will vary depending on the individual PCB congener and are specified in the published method (EPA, 2010). EPA method 1668C has specific criteria for method blanks that must be met (see Section 9.5.2 of the published method). Table 6 of the published EPA method 1668C lists the percent recovery for individual PCB congeners. MQOs falling outside of the acceptance limits will be reviewed by the Project Manager for their usability.

Table 3. MQOs for Laboratory Analyses

Analyte	Bias		Precision		Instrument Performance	Method Blank [#]
	Lab Control Sample ⁺ (recovery)	Matrix Spikes (recovery)	Lab Duplicates (RPD) [*]	Matrix Spike Duplicates (RPD) [*]	Surrogate Standards ⁺ (recovery)	
209 PCB Congeners	15- 145%	60 - 140%	< 50%	< 50%	5-145%	< 1.0 ppb ⁺⁺
Titanium	85-115%	75-125%	≤ 20%	≤ 20%	---	< 1.0 ppm ^{**}

⁺For PCB Congeners: EPA Method 1668C laboratory control samples (which is OPR for ongoing precision and recovery in the method) and surrogate standards are listed in Table 6 of the published method for individual congeners (EPA, 2010).

^{*}RPD = relative percent difference

[#]For PCB Congeners: EPA Method 1668C blank criteria is described in Section 9.5.2 of the published method.

⁺⁺ppb = parts per billion

^{**}ppm = parts per million

7.0 Experimental Design

7.1 Study Design

An estimated 60 samples from the proposed product categories (about 12 samples from each category) will be selected for PCB congener analysis. Ecology staff will coordinate with contract officers from DES to collect products purchased for use by state agencies. Products will be brought back to Ecology headquarters, separated into individual components, screened using an XRF analyzer, and sent to a laboratory for analysis of PCB congeners.

The XRF screening is used to collect information that may help associate particular analytes involved in the manufacturing process of products (like titanium) with the presence of PCBs. For example, larger molecular weight PCBs (PCB-206, PCB-208, and PCB-209) are produced as a byproduct from the manufacture of the white pigment titanium dioxide (Rodenburg, 2012).

Comparing XRF results, like titanium, with PCB congener results may demonstrate XRF screening to be a useful tool for selecting products in future studies for PCB analysis. To validate XRF screening results for titanium, an estimated 6 samples with detectable levels will be sent to the laboratory for quantitative analysis of titanium.

The XRF results will not be used for the primary selection of samples to be tested in this study. Component samples will be selected for laboratory analysis based on information about the manufacturing process that may identify potential products contaminated with PCBs. Sources of information may include the specific product's Safety Data Sheet (SDS), product labels, [National Institute of Health's Household Product Database](#), [International Living Future Institute's Declare Product Database](#), and published literature review.

8.0 Sampling Procedures

8.2 Containers, Preservation Methods, Holding Times

Laboratory samples will be stored in certified clean wide-mouth 8 oz. glass jars. Low-level sampling techniques will be followed to reduce potential contamination of the samples. Samples anticipated to be sensitive to light degradation (like vehicle and ferry lubricants) will have aluminum foil wrapped around the sample jars. The samples will be packed into coolers with the temperature maintained at less than 6 degrees Celsius and shipped to the contract laboratory for analysis.

There are no demonstrated maximum holding times for PCB congeners in products or other sample matrices. Following EPA method 1668C for PCB congener analysis, the aqueous samples may be stored up to one year in the dark at less than 6 degrees Celsius, solid samples stored up to one year in the dark at less than -10 degrees Celsius, and all sample extracts stored for up to one year in the dark at less than -10 degrees Celsius (EPA, 2010).

8.4 Equipment Decontamination

All tools used in the preparation of samples will be decontaminated following the low-level tool cleaning procedure outlined in Ecology's Product Testing Program *Standard Operating Procedures (SOP) for Sample Collection and Processing, Version 1.0* (SOP PTP001, available upon request).

9.0 Measurement Methods

9.2 Lab Procedures Table

Samples will be sent to a contract laboratory for extraction and analysis using EPA method 1668C for PCB congener analysis (EPA, 2010). Extraction and analysis procedures along with the estimated laboratory reporting limit, are listed in [Table 4](#). EPA method 1668C describes several different extraction techniques for samples that may be used in this study depending on the matrix type: soxhlet/dean stark (SDS) extraction for solids and solid-phase extraction (SPE) or separatory funnel liquid-liquid extraction for liquids or oils. The extraction procedure specific to the sample matrix must be used that will achieve maximum recovery of the extractant. Method quality control procedures and the contract laboratory's approved standard operating procedure (SOP) must be followed. The contract laboratory must contact the Project Manager regarding any analysis issues before proceeding further with the analysis. Any corrective action or deviation from the approved QAPP and procedure must be approved by the Project Manager.

MEL will analyze samples selected for titanium analysis based on XRF screening following the digestion and analysis methods listed in Table 4.

Table 4. Laboratory Methods and Reporting Limits

Analyte	Expected Range of Results ⁺	Matrix	Reporting Limit [*]	Extraction or Digestion Method	Analysis Method	Analysis Instrument
209 PCB Congeners	< 1.0 ppb [#] to 2.3 ppm ⁺⁺	Solids, Oils, Liquids	1.0 ppb [#]	EPA 3540, EPA 3535, EPA 3510	EPA 1668C	HRGC/ HRMS ^{**}
Titanium	<1.0 ppm ⁺⁺ to 500 ppm ⁺⁺	Solids, Oils, Liquids	1.0 ppm ⁺⁺	EPA 3052	EPA 6020	ICP-MS ^{##}

⁺For PCB Congeners: expected range of results based on data from a previous study (Ecology 2016b).

^{*}Actual detection limits and quantitation levels are dependent on the levels of interferences and laboratory background levels rather than instrumental limitations. The reporting limit may vary by congener.

[#]ppb = parts per billion

⁺⁺ppm = parts per million

^{**}HRGC/HRMS = high resolution gas chromatography/high resolution mass spectrometry

^{##}ICP-MS = inductively coupled plasma-mass spectrometry

10.0 Quality Control Procedures

10.1 Table of Lab Quality Control Required

Table 5 displays the minimum laboratory quality control (QC) samples required for PCB and titanium analysis. Laboratory QC tests will consist of laboratory control samples (LCS), sample duplicates, matrix spikes, matrix spike duplicates, surrogates, method blanks and rinseate blanks (if applicable).

Table 5. Laboratory Quality Control Tests and Frequency.

Analyte	Lab Control Samples	Sample Duplicates	Matrix Spikes	Matrix Spike Duplicates	Surrogates	Method Blanks	Rinseate Blanks ⁺
209 PCB Congeners	1/batch	1/batch	1/batch	1/batch	every sample	1/batch	1/batch
Titanium	1/batch	1/batch	1/batch	1/batch	---	1/batch	---

Batch = 20 or fewer samples

⁺Applicable only when including rinseate blanks for assessing contamination from sample processing procedures like cryomilling.

11.0 Data Management Procedures

11.2 Lab data package requirements

The contract laboratory will provide an EPA Level 4 data package in electronic format. The package must include all raw data and quality assurance and control documentation that would be needed to perform an independent review of the results including benchsheets, calibration reports, chromatograms and spectra for all calibration standards and samples.

15.0 References

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