

Quality Assurance Project Plan

Product Testing Program Version 1.0

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Publication Information

Each study conducted by the Washington State Department of Ecology (Ecology) must have an approved Quality Assurance Project Plan. The plan describes the objectives of the study and the procedures to be followed to achieve those objectives. After completing the study, Ecology will post the final report of the study to the Internet.

This Quality Assurance Project Plan is available on Ecology's website at https://fortress.wa.gov/ecy/publications/SummaryPages/1603113.html

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Quality Assurance Project Plan

Product Testing Program Version 1.0

June 2016

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EAP: Environmental Assessment Program

HWTR: Hazardous Waste and Toxics Reduction Program

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2.0 Abstract

The Washington State Department of Ecology (Ecology) primarily conducts product testing studies to ensure compliance with Washington's laws and reporting requirements. Studies may also serve to provide information for understanding sources of toxics entering our environment, to identify potential health risks, and for supporting rulemaking efforts.

It is Ecology policy (Executive Policy 22-01) to have an approved Quality Assurance Project Plan (QAPP) for all Agency-sponsored studies and activities generating and/or interpreting data. This universal QAPP will serve as a plan describing the common practices, procedures, and quality requirements for product testing studies at Ecology. QAPP addenda will be prepared for each individual study to document study-specific goals and technical aspects, including planning, implementation, and data assessments where they differ from the standard plan guidelines presented in this document.

3.0 Background

The Product Testing Program was developed at Ecology to study and address toxic chemicals in products available to and used by Washington residents and businesses. These product studies are developed for a variety of reasons: to assess compliance with current regulations; to investigate priority chemicals and emerging chemicals of concern; to provide recommendations in the development of new legislation or rules; or for other toxic-related efforts.

Several Washington State laws, rules, and executive orders (regulations¹) set limits, restrict presence, or require reporting of the amounts of toxic chemicals in products. Section 3.1.5 provides a list of those regulations.

For each of the studies, Ecology purchases products, pre-screens them if appropriate, sends to lab for analysis of toxic chemicals, and writes reports summarizing the lab data. Ecology enforcement officers use that data to take the appropriate enforcement actions with responsible manufacturers and companies.

3.1 Study area and surroundings

All products available to Washington residents and businesses are subject to Washington State regulations. Products will be considered for study assessments if they are sold in any physical location (e.g., discount stores, department stores, supermarkets, and warehouse clubs) within Washington or if they are accessible for purchase online by Washington residents or businesses.

3.1.1 Logistical problems

Limits on the selection of products available during set product purchasing events (sampling) may require widening parameters of product collection or adding additional sampling events.

Possible underreporting of concentrations from laboratory analyses may occur due to the use of conventional extraction techniques or digestion. The specific nature of individual product matrix may also affect the ability to detect and/or quantitate the analytes of concern. Specialized extraction and digestion techniques – such as supercritical fluid extraction (SFE), microwave-assisted extraction (MAE), microwave-assisted digestion, and accelerated solvent extraction (ASE) – may be of increased applicability in processing product matrices. Extraction and analysis methods will be discussed and/or covered in lab-specific standard operating procedures (SOPs) cited within subject-specific QAPPs.

Laboratory results could be qualified according to the product testing <u>standard qualifier reporting</u> <u>practices</u> if issues occur.

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¹Hereafter the term "regulations" will be used when collectively referencing Washington State laws, rules, and executive orders.

3.1.2 History of the study area

As discussed in the Section 3.0 and detailed in other sections of this QAPP.

3.1.3 Chemicals of concern

Chemicals of concern include those cited and governed by existing regulations (from the list of current regulations in Section 3.1.5); those classified as persistent, bioaccumulative, and toxic (PBT); and emerging chemicals of concern identified by Ecology or other governing bodies.

Product studies are developed for a variety of reasons: to assess compliance with current regulations, to investigate priority chemicals and emerging chemicals of concern, to provide recommendations in the development of new legislation or rules, or for other toxic-related efforts.

3.1.4 Results of previous studies

Reports from Ecology's previous product testing studies can be reviewed by searching: https://fortress.wa.gov/ecy/publications/UIPages/PublicationList.aspx?IndexTypeName=Topic&NameValue=Product+Testing&DocumentTypeName=Publication

Laboratory data and product information from Ecology's product testing studies is viewable by searching the online database: http://ecyapeem/PTDBPublicReporting

3.1.5 Regulatory criteria or standards

The current regulations are listed in Table 1.

Study data² will be transferred to the appropriate client/enforcement officer for assessment of compliance and to address potential compliance issues. The criteria for requiring assessments of compliance will be addressed in study-specific QAPP addenda.

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² Verified data from laboratory confirmation analyses; X-ray fluorescence (XRF) and Fourier transform infrared spectroscopy (FTIR) data may warrant transfer on a case-by-case basis.

Table 1. Current Regulations.

Regulation	Citation	Restriction
Children Safe Product Act (CSPA)	Chapter 70.240 Revised Code of Washington (RCW) and Chapter 173-333 Washington Administrative Code (WAC)	Prohibits the sale of a children's product, or product component, containing phthalates ³ , lead, or cadmium ⁴ above a specific concentration. Requires manufacturer reporting on the presence of 66 chemicals or classes of chemicals of high concern to children (CHCC; Appendix A) in children's products.
Toxics in packaging (TIP)	Chapter 70.95G RCW	Restricts the combined concentration of four toxic metals ⁵ in all packaging to under 100 parts per million (ppm).
Flame retardants	Chapter 70.76 RCW	Bans or restricts the use of the polybrominated diphenyl class of flame retardants in certain consumer products.
Bisphenol A (BPA) in children's products	Chapter 70.280 RCW	Bans the use of BPA in specific children's products and sports bottles.
Mercury Education and Reduction (MERA)	Chapter 70.95M RCW	Reduces or eliminates mercury use in certain products.
Better Brakes Law	Chapter 70.285 RCW and Chapter 173-901 WAC	Reduces or restricts the use of certain toxic chemicals ⁶ in brake pads and shoes.
Polychlorinated biphenyls (PCBs) in state purchased products	RCW 39.26.280 and RCW 39.26.290	Requires the state to limit purchases of products containing PCB contamination.
Copper in antifouling paints	Chapter 70.300 RCW	Over time, reduces copper used as an antifoulant in applications on recreational water vessels.
Lead in wheel weights	Chapter 70.270 RCW	Bans the use of lead and chemicals on the PBT list (WAC 173-333) in automotive wheel weights.
Coal tar sealants	Chapter 70.295 RCW	Restricts the level of polycyclic aromatic hydrocarbons in coal tar pavement sealants.
Flame Retardants in Children's Products	RCW 70.240 and HB 2545	Bans the use of five flame retardants ⁷ in children's products and furniture.
Persistent, Bioaccumulative Toxic (PBT) Chemicals and Metals of Concern	Chapter 70.105 RCW, Chapter 173-333 WAC, Executive Order 04-01	Chemicals or chemical groups meeting or exceeding the criteria for persistence, bioaccumulation, and toxicity criteria (Appendix A).

³ Phthalates: dibutyl phthalate (DBP)), butyl benzyl phthalate (BBP), di-2-ethylhexyl phthalate (DEHP), di-n-octyl phthalate (DnOP), diisodecyl phthalate (DIDP), diisononyl phthalate (DINP).

⁴ Levels of phthalates cannot exceed 1,000 parts per million (ppm), individually or in combination, and lead and cadmium levels are prohibited above 90 ppm and 40 ppm, respectfully.

⁵ Metals: lead, mercury, cadmium, and hexavalent chromium.

⁶ Copper, asbestos, hexavalent chromium, mercury, cadmium, and lead.

⁷ Decabromo-diphenyl ether (deca-BDE), additive tetrabromo-bisphenol A (TBBPA), hexabromo-cyclododecane (HBCD), tris(2-chloroethyl) phosphate (TCEP), and tris(1,3-dichloro-2-propyl) phosphate (TDCPP)

4.0 Study Description

Ecology regularly conducts studies on products to assess compliance with current regulations. Investigations on priority chemicals and emerging chemicals of concern may also be designed to provide recommendations in the development of new legislation or rules.

4.1 Study goals

Studies under the Product Testing Program are carried out to:

- Provide data to Ecology's clients/enforcement officers to assess compliance with applicable Washington State regulations.
- Gather data to help understand sources of toxics entering our environment, to identify potential health risks, and provide information to support rulemaking efforts.

4.2 Study objectives

To meet study goals, Product Testing staff will carry out the following objectives:

- Purchase products available for sale in Washington State.
- Analyze select products for target chemicals.
- Assess compliance to existing regulations and/or further our understanding of the uses of toxic chemicals in products.
- Document study findings.

4.3 Information needed and sources

Existing studies, methods, and data from sources such as the U.S. Consumer Product Safety Commission, U.S. Environmental Protection Agency, European Union and Danish Environmental Protection Agency, as well as peer-reviewed journal articles will be reviewed, as applicable.

- Reviews of existing product testing data will be completed to help provide a basis for:
 - o study scoping
 - o chemical selection
 - o product selection
 - o method development
- For Children's Safe Product Act (CSPA) and Better Brakes related studies, a review prior to product collection of the CSPA database or Better Brakes database will help guide selection of retailers and products to target.

4.4 Target population

Studies will be performed on products accessible to Washington residents, businesses, and through state procurement.

4.5 Study boundaries

Products will be obtained by direct purchasing from retail stores within Washington, via online retailers, and from state-purchased sources.

4.6 Tasks required

Table 2 outlines key roles and responsibilities.

To meet study goals, Product Testing staff will carry out the following tasks:

- Purchase products for evaluation of target chemicals.
- Record product information on purchased products in Ecology's Product Testing Database (PTDB).
- Separate products into product components and catalog the components in the PTDB.
- Screen product components, as applicable, utilizing the X-ray fluorescence (XRF) and/or Fourier transform infrared spectroscopy (FTIR) instruments.
- Select samples (e.g., product components) for laboratory analysis.
- Prepare and submit samples for laboratory analysis of target chemicals.
- Analyze and review study data.
- Document study findings.

4.7 Practical constraints

Practical constraints are not anticipated.

4.8 Systematic planning process

This QAPP, and a study-specific addendum, represents adequate systematic planning for each study.

5.0 Organization and Schedule

5.1 Key roles and their responsibilities

Table 2. Roles and Responsibilities (see Product Testing Charter for more details).

Role	Responsibilities
HWTR Product Testing Management Lead	Reviews and approves scopes and budgets for all projects. Provides review of all draft plans and approves all final QAPPs. Reviews all drafts and approves all final reports. Coordinates inter-program efforts.
RTT Toxics Policy Coordinator	Advises study proponents as needed. Reviews and approves external communications of study results.
Client:	 Within respective subject matter field: Proposes studies and clarifies project scope. Provides review of draft plans and approves final QAPPs. Reviews drafts and approves final reports.
EAP and HWTR Project Managers	Researches study area and writes QAPPs, QAPP addenda, and reports. Coordinates with laboratories to obtain analytical services. Conducts QA review of data, analyzes and interprets data. Guides assistants in various roles and tasks. Provides peer reviews.
Sampling and Processing Lead; Project Assistant	Performs product purchasing events, login, XRF & FTIR screening, sample preparation, and PTDB records review. Oversees temporary staff/interns' tasks. Provides review of all draft plans.
EAP Product Testing Management Lead	Management coordinator for EAP. Provides internal review of EAP QAPPs. Provides review of all draft plans and reports, and approves all final QAPPs and reports. Helps coordinate inter-program and efforts as needed.
EAP and HWTR Section Managers	Provides review of draft plans and reports, and approves final QAPPs and reports. Helps coordinate inter-program efforts as needed.
MEL Laboratory Director	Provides review of all draft plans and approves all final QAPPs involving in-house analyses, and through MEL contracting and data review.
HWTR Quality Assurance Representative	Provides review of all HWTR draft plans, and approves all final HWTR QAPPs. Provides review of reports, when requested.
EAP Quality Assurance Representative	Provides review of all EAP draft plans, and approves all final EAP QAPPs. Provides review of reports, when requested.

EAP: Environmental Assessment Program HWTR: Hazardous Waste and Toxics Reductions Program

MEL: Manchester Environmental Laboratory MERA: Mercury Education and Reduction Act

PTDB: Product Testing Database QAPP: Quality Assurance Project Plan

TIP: Toxics in Packaging RTT: Reducing Toxic Threats

5.2 Special training and certifications

Ecology staff conducting sample processing and screenings will be trained according to the SOPs listed in Section 8.1.

Staff will follow and participate in all required agency and program health and safety trainings:

- Program Safety Plans and trainings.
- Agency purchasing and contracts training, as applicable.
- Agency Medical monitoring, as applicable.

5.3 Organization chart

Table 2 lists the key roles and responsibilities.

5.4 Study schedule

The annual product testing program plan outlines the timeline for studies funded in each year as outlined in the Product Testing Program Charter.

Within each study, a schedule for completing product collection and laboratory work, data entry and data review of the PTDB, and report deadline is essential for implementing a study plan. Individual project schedules will be outlined in QAPP addenda.

In general, for larger or more complex studies more time will be needed to complete the study (e.g., external lab contracting, online purchasing or large sample numbers). The project manager should consult the clients, assistants, and the laboratories performing the analyses to assign reasonable dates for specific activities.

5.5 Limitations on schedule

Limitations on sampling staff, processing staff, instrumentation, MEL availability, etc., will be taken into account during development of the annual product testing plan (see Product Testing Charter for more details). Limitations that cannot be accommodated are not anticipated.

At Ecology Headquarters (HQ), processing staff, instrumentation, and resources will be tracked on the <u>Product Testing SharePoint calendar</u> to limit potential study conflicts and delays.

5.6 Budget and funding

Product Testing Program studies will be carried out using available funding as allocated by the HWTR program or other Ecology programs.

Individual study budgets will be allocated during the annual product testing planning based on available funding (see Product Testing Charter for more details). Each study budget will be preestablished after thorough collaborative scoping by the Product Testing management, project managers, and other staff as necessary. Initial scoping will include best estimates of sample types and quantities. Final sample types and quantities will be solidified in QAPP addenda.

Individual study QAPP addenda will describe the budget and funding for specific activities such as sampling (product collection), lab analyses, and any other contractual services (e.g., cryomilling, data validation, and other specialized services).

6.0 Quality Objectives

6.1 Decision quality objectives

Decision quality objectives (DQOs) are not anticipated.

6.2 Measurement quality objectives

The following target measurement quality objectives (MQOs) are based upon those obtained in previous studies:

Tables 3a-f. Measurement Quality Objectives.

3a. Metals

Analyta	Bi	as	Prec	ision	Extraction and Instrument Performance	Reporting
Analyte	LCS (% recov.)	Matrix Spikes (% recov.)	Lab Duplicates (RPD)	Matrix Spike Duplicates (RPD)	Surrogate Standards (% recov.)	Limit
Metals	85 - 115%	75 - 125%	≤ 20%	≤ 20%	n/a	1.0 ppm
Mercury	90 - 110%	80 - 120%	≤ 20%	≤ 20%	n/a	0.0020 ppm

3b. Phthalates

Analyta	Bi	as	Pre	cision	Extraction and Instrument Performance	nce Reporting Limit ds 7.)
Analyte	LCS (% recov.)	Matrix Spikes (% recov.)	Lab Duplicates (RPD)	Matrix Spike Duplicates (RPD)	Surrogate Standards (% recov.)	Limit
Phthalates ^a	50 - 150%	50 - 150%	≤ 40%	≤ 40%	50 - 150%	5.0 ppm

^aPhthalates: DEHP, BBP, DEP, DnHP, DIDP, DINP, DMP, DBP, and DnOP.

3c. Parabens

Analyta	Bia	as	Pre	cision	Extraction and Instrument Performance	Reporting Limit	
Analyte	LCS (% recov.)	Matrix Spikes (% recov.)	Lab Duplicates (RPD)	Matrix Spike Duplicates (RPD)	Surrogate Standards (% recov.)	Limit	
Parabens#	60 - 140%	60 - 140%	≤ 20%	≤ 20%	70 - 140%	5.0 ppm	

^{*}Parabens: methyl paraben, ethyl paraben *n*-propyl, and butyl paraben: *n*-butyl paraben (CAS 94-26-8) and *iso*-butyl paraben (4247-02-3).

3d. Other CSPA Chemicals

Analyte	Bias		Pred	cision	Extraction and Instrument Performance	Reporting
·	LCS (% recov.)	Matrix Spikes (% recov.)	Lab Duplicates (RPD)	Matrix Spike Duplicates (RPD)	Surrogate Standards (% recov.)	Limit
Formaldehyde	50 - 150%	50 - 150%	≤ 40%	≤ 40%	n/a	5.0 ppm
Benzene	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
Vinyl chloride	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
Methylene chloride	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
Carbon disulfide	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	10 ppm
Methyl ethyl ketone (MEK)	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
1,1,2,2-Tetra- chloroethane	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
Bisphenol A (BPA)	60 - 140%	40 - 140%	≤ 30%	≤ 30%	25 - 150%	1.0 ppm
Hexa- chlorobutadiene	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	30 ppm
Ethylbenzene	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
Styrene	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
4-Nonylphenol ¹	50 - 150%	50 - 150%	≤ 40%	≤ 40%	50 - 150%	50 ppm
Acrylonitrile	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
Ethylene Glycol	50 - 150%	50 - 150%	≤ 40%	≤ 40%	n/a	20 ppm
Toluene	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
1, 4-Dioxane	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	1.0 ppm
D4 ²	70 - 125%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	50 ppm

¹ 4-Nonylphenol branched (CAS 84852-15-3) and 4-Nonylphenol (linear; CAS 104-40-5) ² D4 = Octamethylcyclotetrasiloxane

3e. Flame Retardants

	В	ias	Precision		Extraction and Instrument Performance	Paparting	
Analyte	LCS (% recov.)	Matrix Spikes (% recov.)	Lab Duplicates (RPD)	Matrix Spike Duplicates (RPD)	Surrogate Standards (% recov.)	Reporting Limit	
Decabromo- diphenyl ether (deca-BDE)	50 - 150%	50 - 150%	≤ 40%	≤ 40%	50 - 150%	100 ppm	
Tetrabromo- bisphenol A (TBBPA)	60 - 140%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	100 ppm	
Hexabromo- cyclododecane (HBCD)	60 - 140%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	100 ppm	
tris(2-chloroethyl) phosphate (TCEP)	60 - 140%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	100 ppm	
tris(1,3-dichloro-2- propyl) phosphate (TDCPP)	60 - 140%	60 - 140%	≤ 40%	≤ 40%	50 - 150%	100 ppm	

3f. Polychlorinated Biphenyls (total PCBs)

Analyta	Bia	as	Pre	ecision	Extraction and Instrument Performance	Reporting
Analyte	LCS (% recov.)	Matrix Spikes (% recov.)	Lab Duplicates (RPD)	Matrix Spike Duplicates (RPD)	Surrogate Standards (% recov.)	Limit
PCBs ^ø	15 - 115%	60 - 140%	≤ 50%	≤ 50%	15 - 150%	0.5 ppb#

^øPCB congeners-low level, individual.

6.2.1 Targets for precision, bias, and sensitivity

6.2.1.1 Precision

Precision is a measure of the variability in the results of measurements due to random error. Laboratory precision will be assessed through laboratory duplication of product samples. Submission of field duplicates to assess the variability of the sample processing procedure may be warranted for individual studies. See Tables 3a-f for MQOs.

[#]Based upon detection limits and quantitation levels which are dependent on the levels of interferences and laboratory background levels rather than instrumental limitations. The reporting limit may vary by congener.

6.2.1.2 Bias

Bias is the difference between the population mean and the true value. Assessments of laboratory bias will be determined by analysis of laboratory control samples (LCSs), matrix spiked samples, and standard reference materials. See Tables 3a-f for MQOs.

6.2.1.3 Sensitivity

Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest.

Reporting Limits for each analyte are listed in Tables 3a-f.

6.2.2 Targets for comparability, representativeness, and completeness

6.2.2.1 Comparability

Comparability will be ensured by implementing standardized procedures for sampling and analysis.

Appropriate established methods, procedures, and SOPs listed in Section 8.1 will be followed as applicable by matrix and analyte.

Within an individual study, all laboratories performing the same analysis should be required to meet the similar MQOs and use similar QC acceptance criteria when possible.

6.2.2.2 Representativeness

Within each study's boundaries Ecology staff will purchase products representative of those available to Washington residents.

A variety of Washington retailer locations (e.g., discount stores, department stores, supermarkets, and warehouse clubs) will be visited to obtain a wide assortment of products. The practice of purchasing products online will also be employed to acquire additional products accessible to most Washington residents. When appropriate, products will also be obtained from state procurements.

6.2.2.3 Completeness

The project manager will consider the study to have achieved completeness if 95% of the laboratory samples are analyzed acceptably.

7.0 Sampling Process Design (Experimental Design)

7.1 Study design

Products will be purchased from retail stores, online through internet retailers, and from state agency procurements. For many studies, products generally should be collected in amounts significantly greater than the anticipated numbers of samples to be submitted for laboratory analysis. For example, 200-300 products⁸ should be sufficient for many studies when the laboratory sample numbers will be 50-75 per analyte.

Products will be brought back to Ecology HQ, isolated into separate components, and cataloged in the PTDB. As applicable, product components will be screened for metals, chlorine and bromine using an XRF analyzer, and for phthalates or other targeted compounds using the FTIR.

Component samples will be selected for laboratory analysis based on XRF and FTIR screenings, literature reviews, and review of previous study data or external databases (Section 4.3).

Sample numbers and target analytes for laboratory analyses are defined in each addendum.

7.1.1 Field measurements

Not applicable.

7.1.2 Sampling location and frequency

Products will be purchased from retailers, online retailers, and state agency procurements during a multi-week period upon completion of an approved QAPP addendum.

7.1.3 Parameters to be determined

A list or table of parameters/analytes to be determined will be included in each study QAPP addendum.

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⁸ An individual product is often made up of numerous components. Alternatively, only one component from a product may be targeted. Additional rationale may require adjustments to product, sample numbers, or both within each study.

7.3 Assumptions underlying design

For sampling (purchasing), the practice of statewide distribution by retail chain stores assumes that products purchased from one store are typical of products sold by the same retail chain store at other locations throughout the state.

Products purchased online are available equally to most in Washington State due to the wide accessibility of the Internet.

7.4 Relation to objectives and site characteristics

Not applicable.

7.5 Characteristics of existing data

Previous QAPPs and reports can be found by searching Product Testing publications: https://fortress.wa.gov/ecy/publications/UIPages/PublicationList.aspx?IndexTypeName=Topic&NameValue=Product+Testing&DocumentTypeName=Publication.

8.0 **Sampling Procedures**

Sampling and measurement SOPs 8.1

Product collection, cataloging, and preparation will follow Ecology's Product Testing SOPs⁹:

- Product Testing Sample Collection and Sample Processing draft
- Product Testing Data Entry and Database draft

Component sample screening by the XRF and FTIR will follow the respective SOPs.

- X-ray Fluorescence (XRF) Screening Consumer Products draft
- Fourier Transform Infrared Spectroscopy (FTIR) Screening Consumer Products draft

Containers, preservation methods, holding times 8.2

Products and product components will be stored according to the procedures described in the Sample Collection and Sample Processing SOP.

Laboratory samples will be stored in certified wide-mouth 4 oz. or 8 oz. glass jars with no preservation. Jars can be ordered through the MEL, using the Sample Container Request Form.

No holding times or preservation methods have been established for product matrices. Where there is concern of the possible effects of volatilization, temperature, or light sensitivity, laboratory staff should be consulted and specific conditions addressed in each study QAPP addenda.

Invasive species evaluation 8.3

Not applicable.

Equipment decontamination 8.4

To obtain reliable and usable data, it is essential to be conscious of and employ effective decontamination processes.

A combination of many factors must be considered to select an appropriate method of decontamination. The target analytes, required reporting limit, and enforcement level of each analyte in the sampled media should be considered. Decontamination procedures should follow protocols outlined in the Sample Collection and Sample Processing SOP.

⁹ At the time of the publication of this document, the Product Testing SOPs are actively being formalized in the Ecology SOP format. Current accepted standard protocols will be followed during the SOP updating processes.

8.5 Sample ID

Individual product component IDs are auto-generated by the PTDB during product and component login, as described in the Product Testing Data Entry and Database SOP. Product component IDs combine information from store of purchase, purchase event, product, and component of product (e.g., "TG-1-1-2" = Target, purchase event 1, product 1, 2nd component of the product tested).

For samples sent to the MEL, submit the <u>Pre-Sampling Notification</u> form at least three weeks prior to the planned submission of samples. MEL will generate a seven-digit work order number (WO#; e.g., 1601027) for each sample set(s) for an individual study. During HQ sample processing, the addition of a two-digit suffix to the WO# will result in a laboratory sample ID number (e.g., 1601027-01, 1601027-02) for each sample.

Sample ID numbers and their corresponding product components sample ID (PTDB generated) are recorded on both the sample containers and the Chain-of-Custody form.

Samples that are contracted through MEL will be assigned sample IDs in the same manner as samples to be analyzed by MEL.

8.6 Chain of custody

Chain of custody will be maintained for all samples throughout all studies. Products and product components from samples requiring enforcement will be held under chain of custody until enforcement activities have been completed. Appropriate Enforcement Officers will be consulted prior to any product, component, and sample disposal.

8.7 Field log requirements

Advertisements, photos of product marketing, and other information gathered during study purchasing events will be recorded and uploaded or scanned into the PTDB by study. Specific protocols are outlined in the Sample Collection and Sample Processing SOP.

8.8 Other activities

Necessary activities are detailed in other sections of this QAPP.

9.0 Measurement Methods

9.1 Field procedures table/field analysis table

Not applicable.

9.2 Lab procedures table

The following laboratory procedures and methods are based upon those obtained in <u>previous studies</u>. When known, matrices requiring variations to reporting limits, preparation, or analytical methods are delineated. Tables 4a-f contain guidance for the standard accepted methods used within this program. Other analytes or analysis methods not listed in the tables below may be used, provided the appropriate method accreditation requirements (Section 9.5) are met. Additional analytes or analysis methods will be documented in study-specific QAPP addenda.

Tables 4a-f. Laboratory Methods and Reporting Limits.

4a. Metals

Analyte	Expected Range of Results	Matrix	Reporting Limit	Preparation Method	Analysis Method	Analysis Instrument
Metals*^	<1 - 1,000 ppm	Solids*	1.0 ppm	EPA 3052 mod+	EPA 6020	ICP-MS
Mercury [∆]	<2 – 100 ppb	Solids*	2 ppb	n/a	EPA 7473	DMA-AAS

^{*}CSPA metals suite: antimony, arsenic, cadmium, cobalt, molybdenum, mercury, and lead.

4b. Phthalates

Analyte	Expected Range of Results	Matrix	Reporting Limit (ppm)	Preparation Method	Analysis Method	Analysis Instrument
Phthalates ^a <5 -	.5, 50,000	Solids*	5.0 - 50	EPA 3546 mod or CPSC- C1001-09.3	EPA 8270D mod	GC-MS
	<5 - 50,000 ppm	Plastic	5.0 - 50 ^b	EPA 3546 mod or CPSC- C1001-09.3	EPA 8270D mod	GC-MS

^aPhthalates suite: DEP, DBP, DnHP, BBP, DEHP, DnOP, DIDP, DINP, and DMP.

[^]Packaging metals: chromium, cadmium, lead, and mercury.

 $^{^{\}Delta}$ When low-level mercury analysis is necessitated, additional clean preparation techniques will be discussed in project-specific QAPP addenda.

⁺MEL's EPA 3052 mod. method omits hydrofluoric acid (HF). Glass matrices require the use of HF and will be analyzed by a contract laboratory.

^{*}Solids is a broad category to include most material matrices that are not liquids or gases (e.g., plastic, foam, metal, fabric).

^bIndividual reporting limits may vary based upon analyte and specific plastic type.

^{*}Solids is a broad category to include most material matrices that are not liquids or gases (e.g., foam, metal, fabric).

4c. Parabens

Analyte	Expected Range of Results	Matrix	Reporting Limit (ppm)	Limit Preparation Method		Analysis Instrument
Parabens#	<5 - 500 ppm	liquid, cream, gel	5.0	EPA 3580A	EPA 8321A	HPLC-MS

^{*}Parabens: methyl paraben, ethyl paraben *n*-propyl, and butyl paraben: *n*-butyl paraben (CAS 94-26-8) and *iso*butyl paraben (4247-02-3).

4d. Other CSPA Chemicals

Analyte	Expected Range of Results	Matrix	Reporting Limit (ppm)	Preparation Method	Analysis Method	Analysis Instrument
Formaldehyde	<5 - 500 ppm	Solids*	5.0	n/a	EPA 8315A or 6850 mod.	HPLC or LCMS
Benzene	<1 - 500 ppm	Solids*	1.0	EPA 5030B	EPA 8260C	GC-MS
Vinyl chloride	<1 - 500 ppm	Solids*	1.0	EPA 5030B	EPA 8260C	GC-MS
Methylene chloride	<1 - 500 ppm	Solids*	1.0	EPA 5030B	EPA 8260C	GC-MS
Carbon disulfide	<10 - 500 ppm	Solids*	10	EPA 5030B	EPA 8260C	GC-MS
Methyl ethyl ketone (MEK)	<1 - 1,000 ppm	Solids*	1.0	EPA 5030B mod	EPA 8260C	GC-MS
1,1,2,2-Tetra- chloroethane	<1 - 500 ppm	Solids*	1.0	EPA 5030B	EPA 8260C	GC-MS
Bisphenol A (BPA)	<1 - 100 ppm	Solids*	1.0	lab specific	EPA 1694	LC/MS/MS
Hexa- chlorobutadiene	<30 - 500 ppm	Solids*	30	EPA 5030B	EPA 8260C	GC-MS
Ethylbenzene	<1 - 500 ppm	Solids*	1.0	EPA 5030B	EPA 8260C	GC-MS
Styrene	<1 - 1,000 ppm	Solids*	1.0	EPA 5030B	EPA 8260C	GC-MS
4-Nonylphenol ¹	<50 - 1,000 ppm	Solids*	50	EPA 3546	EPA 8270D	GC-MS
Acrylonitrile	<1 - 500 ppm	Solids*	1.0	EPA 5030B	EPA 8260C	GC-MS

Analyte	Expected Range of Results	Matrix	Reporting Limit (ppm)	Preparation Method	Analysis Method	Analysis Instrument
Ethylene glycol	<20 - 1,000 ppm	Solids*	20	n/a	EPA 8015C or 8260 mod.	GC-FID or GC-MS
Toluene	<1 - 500 ppm	Solids*	1.0	EPA 5030B	EPA 8260C	GC-MS
1, 4-Dioxane	<1 - 500 ppm	Solids*	1.0	EPA 5030B	EPA 8260C	GC-MS
D4 ²	<50 - 1,000 ppm	Solids*	50	lab-specific	lab-specific	GC-MS

¹ 4-Nonylphenol branched (CAS 84852-15-3) and 4-Nonylphenol (linear; CAS 104-40-5).

4e. Flame Retardants

Analyte	Expected Range of Results	Matrix	Reporting Limit (ppm)	Preparation Method	Analysis Method	Analysis Instrument
Decabromo- diphenyl ether (deca-BDE; PBDE 209)	< 100 -100,000 ppm	Solids*	100	EPA 3546	EPA 8270	GC-MS
Tetrabromo- bisphenol A (TBBPA)	< 100 -100,000 ppm	Solids*	100	EPA 3540C	EPA 1694	LC/MS/MS
Hexabromo- cyclododecane (HBCD)	< 100 -100,000 ppm	Solids*	100	EPA 3540C	EPA 1694	LC/MS/MS
tris(2- chloroethyl) phosphate (TCEP)	< 100 -10,000 ppm	Solids*	100	EPA 3540C or 3546	EPA 8270	GC-MS
tris(1,3- dichloro-2- propyl) phosphate (TDCPP)	< 100 -100,000 ppm	Solids*	100	EPA 3540C or 3546	EPA 8270	GC-MS

^{*}Solids is a broad category to include most material matrices that are not liquids or gases (e.g., plastic, foam, metal, fabric).

² Octamethylcyclotetrasiloxane.

^{*}Solids is a broad category to include most material matrices that are not liquids or gases (e.g., plastic, foam, metal, fabric).

4f. Polychlorinated Biphenyls (Total PCBs)

Analyte	Expected Range of Results	Matrix	Reporting Limit	Preparation Method	Analysis Method	Analysis Instrument
PCBs ^ø	<0.5 - 500 ppb	Solids*	0.5 ppb	EPA 1668B	EPA 1668C	HR GC-MS

^øPCB congeners, individual.

9.3 Sample preparation and screening method(s)

See Tables 3a-f for laboratory preparation methods that can meet the acceptance criteria described in Tables 4a-f.

Sample processing and preparation done by HQ staff will follow the procedures outlined in the Sample Collection and Sample Processing SOP. The screening of product and component samples by XRF and FTIR should follow the procedures outlined in the XRF and FTIR SOPs.

The process of pre-processing a sample by means of cryomilling may be necessary for some materials and matrices and heterogeneous samples. Cryomill processes should follow those outlined in a pre-approved laboratory SOP (e.g., MEL SOP 720033).

9.4 Special method requirements

When samples are cryomilled, additional laboratory quality control (QC) procedures will include: (1) rinsing the cryomill vessels with deionized water, reagents, or solvents as specified by laboratory SOPs between each sample and (2) testing one rinse blank per batch of 20 samples processed for all analytes.

Current certified reference materials (CRMs) or standard reference materials (SRMs) should be required for all analyses where practicable. As more specific matrix reference standards become available, they should be acquired and added to sample batches. It is preferable if reference standard analyte concentrations are at or near compliance levels. Current reference materials available are listed in Appendix B.

9.5 Lab(s) accredited for method(s)

Accreditation assures that the laboratory has demonstrated its capability to reliably generate and report the analytical data (WAC <u>173-50-040</u>, definition of "accreditation"). Laboratories performing analyses must be accredited in the method(s) in appropriately similar matrices types (i.e., solid, liquid) prior to testing product samples. When new methodology is likely

^{*}Solids is a broad category to include most material matrices that are not liquids or gases (e.g., plastic, foam, metal, fabric).

needed for analyses where no guidance or similar methods has been performed, project managers will consult appropriate QA representatives during the QAPP addendum drafting period. As required by Executive Policy 22-02, submit a "Request for Waiver to Required Use of Accredited Lab" Form 070-152 to the Agency QA Officer when it is determined that no labs are appropriately accredited.

10.0 Quality Control Procedures

10.1 Table of lab quality control required

Table 5 outlines the quality control (QC) samples required. QC tests will include at a minimum, one: method blank, laboratory control sample (LCS), duplicate sample, matrix spike and matrix spike duplicate per analysis batch of 20 samples or less. Surrogates will be added and analyzed in each sample as specified in applicable methods and laboratory SOPs. When available, standard reference materials should be included. Additional rinseate blanks (e.g., equipment, cryomill) will be included when such processes are employed to prepare samples.

Table 5. Laboratory Quality Control Tests and Frequency.

Method Blanks	Laboratory Control Samples (LCS)	Laboratory Duplicates	Matrix Spikes	Matrix Spike Duplicates	Surrogates*	Standard Reference Materials**	Rinseate Blanks^
1/batch	1/batch	1/batch	1/batch	1/batch	every sample	1/batch	1/batch

^{*}Not applicable for analyses of metals, mercury, and ethylene glycol.

Batch = 20 or fewer samples.

10.2 Corrective action processes

HQ staff will adhere to appropriate SOPs and study-specific processing and preparation protocols. Where the integrity of the processing and preparation processes are in question, the project manager will determine if samples should be re-sampled, re-analyzed, rejected, or used with appropriate qualification.

The laboratory analysts will document whether project data meet method QC criteria. As soon as it is recognized, the lab will notify the project manager if substantial departures of method techniques will be necessitated. Any departures from normal analytical methods will be documented by the laboratory and described in the case narrative(s).

The project manager will work closely with the laboratories, appropriate QA representatives(s), and any Third Party reviewers conducting data reviews. The project manager will determine whether samples should be re-sampled, re-analyzed, rejected, or used with appropriate qualification.

^{**}When similar matrix material is available.

[^]Applicable only when including rinsate blanks for assessing contamination from sample processing procedures.

11.0 Data Management Procedures

11.1 Data recording/reporting requirements

Study data will be stored in Ecology's PTDB. The database stores product descriptions, purchase receipts, photos of products, screening data, laboratory data, and case narratives. Laboratory data with accompanying product information will be available to the public through an external search application at: https://fortress.wa.gov/ecy/ptdbpublicreporting/.

Laboratory data will be transferred electronically from MEL's Laboratory Information Management System (LIMS) into the PTDB or arrive as an electronic data deliverable (EDD) package.

For all data to be loaded into the PTDB, a QA review (data verification) of both LIMS-delivered and contract EDDs data will be performed by the project manager within 3 weeks of receipt of data. Upon completion of the QA review, the project manager or designated staff will upload the final QA-reviewed data to the internal PTDB. Transfer of data to the client/enforcement officer(s) will occur within one week after the data verification is complete.

Note:

LIMS-delivered laboratory data are transferred by work order to the PTDB and stored in the project manager's Lab batches holding area.

Copies of raw EDD data and MEL LIMS-delivered data with respective case narratives will be saved to the <u>Product Testing SharePoint</u>, stored in an appropriate study folder.

Case narratives for project data are not currently available through the external search. They will be available to the public when requested.

Internally generated screening data

All XRF and FTIR raw data will be initially verified by the analyst for completeness and accuracy (per the applicable SOP), and the data will be made available to project managers prior to the laboratory analysis sample selection process.

Verified XRF screening results are uploaded to the PTDB, are available internally through Lab results searches, and can be exported out into Comma Separated Value (.CSV) files. Raw XRF spectrum/data files are stored in the internal PTDB as .NDT file attachments to uploaded XRF batches. Narratives attached to XRF batches provide a discussion of issues encountered during the XRF screening.

The FTIR data are stored in the <u>Product Testing X Drive</u> as both raw instrument data files (.SPA files) and library identification files in Microsoft Document Writer (.XPS) form. The FTIR data are saved in study-specific folders. The FTIR results are not currently stored within the PTDB.

XRF and FTIR data are used for internal preliminary screening processes only and are not searchable on the external PTDB.

11.2 Lab data package requirements

Laboratories performing analyses under this program will provide an electronic deliverables package after completing their work.

Case narratives will be included to discuss any problems encountered with the analyses, corrective action taken, changes to the requested analytical method, and a glossary for data flags and qualifiers. All sample results and quality control data will be included with the package.

When data validation is required, study-specific contract laboratory requirements will be discussed more thoroughly in contract documents (e.g., Request for Laboratory Services).

Comprehensive data packages should include, but are not limited to:

- data in appropriate <u>EDD format</u> (provided by Ecology)
- signed and dated chain-of-custody paperwork
- sample condition at arrival
- text narratives
- all raw data that must include, but are not limited to:
 - o preparation benchsheets
 - o analytical result reports
 - o analytical sequence (run) logs
 - o chromatograms and spectra for all samples
 - o chromatograms and spectra for all standards (calibration and continuing check) and batch QC samples (matrix spikes, replicates, and blanks)
- additional QA/QC documentation must be provided, including, but not limited to:
 - o standard logs
 - Certificates of Analysis for standards
 - o qualifier and flag descriptions

11.3 Electronic transfer requirements

Case narratives will be in Adobe Acrobat (.PDF) format, and EDDs will be in a .CSV spreadsheet format.

For data generated by MEL, case narratives will be sent to the project manager via email and electronic data will be delivered through LIMS into the internal PTDB.

MEL contracted laboratory data will be submitted back to MEL as a fully paginated and bookmarked comprehensive .PDF file, with all contract-specified content, along with the EDD (.CSV). Smaller files may be sent through email, while larger files may be required to be submitted on compact disk. After performing a review for qualitative and quantitative precision and bias on the data, MEL's QA representative will compose a case narrative of the data review and deliver the case narrative and EDD via email to the project manager. Contract laboratories will be provided with the EDD template and EDD Help documents at the time of the request of services.

11.4 Acceptance criteria for existing data

Not applicable.

11.5 EIM/STORET data upload procedures

Not applicable. Section 11.1 describes the database where data will be stored for this project.

12.0 Audits and Reports

12.1 Number, frequency, type, and schedule of audits

Audits may be established for individual studies to help improve consistency, improve adherence to SOPs, provide a forum for sharing innovations, and strengthen Ecology's data QA program.

Laboratories must participate in performance and system audits of their routine procedures. Results of these audits must be made available on request.

12.2 Responsible personnel

Audits must be performed by a qualified accreditation body (e.g., Ecology's QA Officer or Laboratory Accreditation Unit).

12.3 Frequency and distribution of report

Published reports summarizing the data and findings will be generated in the short report format. A final published short report will include:

- An overview of the study.
- Clear and concise goals and objectives of the study.
- General descriptions of products purchased.
- Results of laboratory analyses and data quality.
- Statistical summaries of laboratory results.
- Summary of laboratory data collected.

12.4 Responsibility for reports

See Table 1 and the Product Testing Charter for descriptions of roles and responsibilities.

For individual studies, the project manager will be responsible for a publicized final report of the study data.

Other forms of study documentation may include web page abstracts, press releases, or technical memos. These forms of study documentation may require a collaborative effort between several staff.

13.0 Data Verification

13.1 Field data and sample process verification, requirements, and responsibilities

The project manager, or assigned and qualified designee, will conduct a final review of product entry and screening data generated within a project.

All data entered into the PTDB will be reviewed by HQ staff at several stages during each study according to the Product Testing – Data Entry and Database SOP.

13.2 Lab data verification

Data verification is a review process to assess the quality and completeness of analytical data. An initial verification of data packages will be performed by qualified laboratory staff experienced with the method. A detailed examination of all lab data sets includes a review for errors, omissions, interpretations, calculations, qualifications, and compliance with all appropriate QC acceptance criteria and contract requirements. Case narratives will be generated by laboratory staff and submitted, along with the lab data, to the project managers. Narratives serve as a summation of laboratory activities and discussion and statement of lab data package quality. The narrative will include a discussion describing if (1) MQOs were met, (2) proper analytical methods and protocols were followed, (3) calibrations and controls were within limits, and (4) data were consistent, correct, and complete, without errors or omissions.

Usage of data flags and qualifiers will be thoroughly examined and verified for appropriateness. When flags are used in contract lab data, the project manager should consult laboratory staff and appropriate QA representatives to ensure the systematic conversion of flags into accepted Product Testing data qualifiers (i.e., E, J, N, NJ, REJ, U, and UJ).

The project manager, with guidance of a QA representative as necessary, will review case narratives and perform data verifications and will decide if further action is necessary. The project manager is responsible for the final acceptance of the study data. Final determination of whether to accept, reject, or accept the results with qualification will be made only after thorough evaluation of the above-mentioned items.

When data that do not follow contractual requirements are received, the project manager can request reprocessing of the samples and data at the lab(s) time and expense.

13.3 Validation requirements, if necessary

In most cases, third-party independent data validation will not be required because of the expense and difficulty of true validation. Level 4 data will be validated using the appropriate EPA National Functional Guidelines (EPA, 2013 and EPA, 2014).

14.0 Data Quality (Usability) Assessment

14.1 Process for determining whether project objectives have been met

The project manager will assess the quality of the data based on case narratives and data packages. Laboratory QC tests will be examined to determine if the lab(s) met MQOs for method blanks, LCSs, duplicates, matrix spike samples, and surrogates when applicable. Reporting limits will be examined to ensure that the contract-defined reporting limit was met.

Further assessments of duplicate and spike performance will be used to evaluate any effects of sample matrix on the data quality.

Blank evaluation will aid in determining contamination, interferences, and precision for samples with low concentrations near analytical detection limits.

<u>Quality control tables</u> will be generated to document the laboratory data in support of determining whether the project objectives have been met.

14.2 Data analysis and presentation methods

The final report will include a statistical summary of the results. Simple summary statistics will be presented in tables. Example summary statistics may include minimum, maximum, median, and frequencies of detection.

Reports will include a link to the study data available on the external database: https://fortress.wa.gov/ecy/ptdbpublicreporting/.

14.3 Treatment of non-detects

Laboratory data will be reported down to the reporting limit, with an associated "U" or "UJ" qualifier for samples below the reporting limit.

14.4 Sampling design evaluation

The number and type of collected samples should be sufficient to meet the objectives of each study.

14.5 Documentation of assessment

Assessments will occur in the final report (i.e., whether followed plan, met objectives, and overall data quality). Other types of documented assessments may include: research and development findings, summaries of non-compliance, and enforcement action taken.

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16.0 Appendices

Appendix A. Chemicals of Interest

List of Chemicals of High Concern to Children (CHCC)

Chemical	CAS Number
Formaldehyde	50-00-0
Aniline	62-53-3
N-Nitrosodimethylamine	62-75-9
Benzene	71-43-2
Vinyl chloride	75-01-4
Acetaldehyde	75-07-0
Methylene chloride	75-09-2
Carbon disulfide	75-15-0
Methyl ethyl ketone	78-93-3
1,1,2,2-Tetrachloroethane	79-34-5
Tetrabromobisphenol A; TBBPA	79-94-7
Bisphenol A	80-05-7
Diethyl phthalate	84-66-2
Dibutyl phthalate	84-74-2
Di-n-Hexyl phthalate	84-75-3
Phthalic anhydride	85-44-9
Butyl benzyl phthalate (BBP)	85-68-7
N-Nitrosodiphenylamine	86-30-6
Hexachlorobutadiene	87-68-3
Propyl paraben	94-13-3
Butyl paraben	94-26-8
2-Aminotoluene	95-53-4
2,4-Diaminotoluene	95-80-7
Methyl paraben	99-76-3
p-Hydroxybenzoic acid	99-96-7
Ethylbenzene	100-41-4
Styrene	100-42-5
4-Nonylphenol; 4-NP and its isomer mixtures including CAS 84852-15-3 and CAS 25154-52-3	104-40-5
para-Chloroaniline	106-47-8
Acrylonitrile	107-13-1
Ethylene glycol	107-21-1
Toluene	108-88-3
Phenol	108-95-2
2-Methoxyethanol	109-86-4
Ethylene glycol monoethyl ether	110-80-5
Tris(2-chloroethyl) phosphate; TCEP	115-96-8
Di-2-ethylhexyl phthalate	117-81-7
Di-n-octyl phthalate (DnOP)	117-84-0

Chemical	CAS Number
Hexachlorobenzene	118-74-1
3,3'-Dimethylbenzidine and Dyes Metabolized to 3,3'-Dimethylbenzidine	119-93-7
Ethyl paraben	120-47-8
1,4-Dioxane	123-91-1
Perchloroethylene	127-18-4
Benzophenone-2 (Bp-2); 2,2',4,4'- Tetrahydroxybenzophenone	131-55-5
4-tert-Octylphenol; 1,1,3,3-Tetramethyl-4-butylphenol	140-66-9
Estragole	140-67-0
2-Ethylhexanoic acid	149-57-5
Octamethylcyclotetrasiloxane	556-67-2
Benzene, pentachloro	608-93-5
C.I. solvent yellow 14	842-07-9
N-Methylpyrrolidone	872-50-4
2,2',3,3',4,4',5,5',6,6'-Decabromodiphenyl ether; Deca-BDE	1163-19-5
Perfluorooctanyl sulphonic acid and its salts; PFOS	1763-23-1
Phenol, 4-octyl-	1806-26-4
2-Ethyl-hexyl-4-methoxycinnamate	5466-77-3
Mercury & mercury compounds including methyl mercury (22967-92-6)	7439-97-6
Molybdenum & molybdenum compounds	7439-98-7
Antimony & Antimony compounds	7440-36-0
Arsenic & Arsenic compounds including arsenic trioxide (1327-53-3) & dimethyl arsenic acid (75-60-5)	7440-38-2
Cadmium & cadmium compounds	7440-43-9
Cobalt & cobalt compounds	7440-48-4
Tris(1,3-dichloro-2-propyl)phosphate; TDCPP	*13674-87-8
Butylated hydroxyanisole; BHA	25013-16-5
Hexabromocyclododecane; HBCD	25637-99-4
Diisodecyl phthalate (DIDP)	26761-40-0
Diisononyl phthalate (DINP)	28553-12-0

^{*}The presence of Tris (1,3-dichloro-2-propyl)phosphate must be reported in all notices required to be filed after August 31, 2014, according to the phase-in schedule in WAC <u>173-334-110(2)</u>.

[Statutory Authority: Chapter <u>70.240</u> RCW. WSR 13-21-123 (Order 12-02), § 173-334-130, filed 10/22/13, effective 11/22/13. Statutory Authority: Chapter <u>70.240</u> RCW, RCW <u>70.240.040</u>. WSR 11-16-008 (Order 09-04), § 173-334-130, filed 7/21/11, effective 8/21/11.]

PBT List

Chemicals listed alphabetically, and by group when applicable	CAS Number
Aldrin	309-00-2
Chlordane	57-74-9
Chlordecone (Kepone)	143-50-0
Dichlorodiphenyltrichloroethane (DDT)	50-29-3
Dieldrin	60-57-1
Endrin	72-20-8
Heptachlor/Heptachlor epoxide	76-44-8/1024-57-3
Hexabromobiphenyl	59536-65-1
Hexabromocyclododecane	25637-99-4
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Methyl mercury	22967-92-6
Mirex	2385-85-5
Pentachlorobenzene	608-93-5
Short-chain chlorinated paraffins	85535-84-8
Tetrabromobisphenol A	79-94-7
Tetrachlorobenzene, 1,2,4,5-	95-94-3
Toxaphene	8001-35-2
Metals of Concern	
Cadmium	7440-43-9
Lead	7439-92-1
Perfluorooctane sulfonates (PFOS)	
Acid	1763-23-1
Ammonium salt	29081-56-9
Diethanolamine salt	70225-14-8
Lithium salt	29457-72-5
Potassium salt	2795-39-3
Polycyclic aromatic hydrocarbons (PAHs)	
3-Methyl chlolanthrene	56-49-5
7H-Dibenzo(c,g)carbazole	194-59-2
Benzo(a)phenanthrene (Chrysene)	218-01-9
Benzo(b)fluoranthene	205-99-2
Benzo(g,h,i)perylene	191-24-2
Benzo(j)fluoranthene	205-82-3

Chemicals listed alphabetically, and by group when applicable	CAS Number	
Benzo(k)fluoranthene	207-08-9	
Benzo(r,s,t)pentaphene	189-55-9	
Dibenzo(a,e)pyrene	192-65-4	
Dibenzo(a,h)pyrene	**189-64-0	
Dibenzo(a,h)acridine	226-36-8	
Dibenzo(a,h)anthracene	53-70-3	
Dibenzo(a,j)acridine	224-42-0	
Fluoranthene	206-44-0	
Indeno(1,2,3-cd)pyrene	193-39-5	
Perylene	198-55-0	
Polybrominated dibenzodioxins and furans		
2,3,7,8-tetrabromodibenzo-pdioxin	50585-41-6	
2,3,7,8-tetrabromodibenzofuran	67733-57-7	
Polybrominated diphenyl ethers		
Pentabromodiphenyl ether	32534-81-9	
Octabromodiphenyl ether	32536-52-0	
Decabromodiphenyl ether	1163-19-5	
Polychlorinated biphenyls (PCBs)		
2,3',4,4',5 Pentachlorobiphenyl	31508-00-6	
2,3,4,4',5 Pentachlorobiphenyl	74472-37-0	
2,3,3',4,4' Pentachlorobiphenyl	32598-14-4	
3,3',4,4',5,5' Hexachlorobiphenyl	32774-16-6	
2,3',4,4',5,5' Hexachlorobiphenyl	52663-72-6	
2,3,3',4,4',5' Hexachlorobiphenyl	69782-90-7	
2,3,3',4,4',5 Hexachlorobiphenyl	38380-08-4	
2,3,3',4,4',5,5' Heptachlorobiphenyl	39365-31-9	
Polychlorinated dibenzo-p-dioxins		
2,3,7,8 Tetrachlorodibenzo-p-dioxin	1746-01-6	
1,2,3,7,8 Pentachlorodibenzo-p-dioxin	40321-76-4	
1,2,3,4,7,8 Hexachlorodibenzo-p-dioxin	39227-28-6	
1,2,3,6,7,8 Hexachlorodibenzo-p-dioxin	**57653-85-7	
1,2,3,7,8,9 Hexachlorodibenzo-p-dioxin	19408-74-3	
1,2,3,4,6,7,8 Heptachlorodibenzo-p-dioxin	35822-46-9	
1,2,3,4,6,7,8,9 Octachlorodibenzo-p-dioxin	3268-87-9	

Chemicals listed alphabetically, and by group when applicable	CAS Number	
Polychlorinated dibenzofurans		
2,3,7,8 Tetrachlorodibenzofuran	51207-31-9	
1,2,3,7,8 Pentachlorodibenzofuran	57117-41-6	
2,3,4,7,8 Pentachlorodibenzofuran	57117-31-4	
1,2,3,4,7,8 Hexachlorodibenzofuran	70648-26-9	
1,2,3,6,7,8 Hexachlorodibenzofuran	57117-44-9	
1,2,3,7,8,9 Hexachlorodibenzofuran	72918-21-9	
2,3,4,6,7,8 Hexachlorodibenzofuran	60851-34-5	
1,2,3,4,6,7,8 Heptachlorodibenzofuran	67562-39-4	
1,2,3,4,7,8,9 Heptachlorodibenzofuran	55673-89-7	
1,2,3,4,6,7,8,9 Octachlorodibenzofuran	39001-02-0	
Polychlorinated naphthalenes		
Trichloronaphthalene	1321-65-9	
Tetrachloronaphthalene	1335-88-2	
Pentachloronaphthalene	1321-64-8	
Hexachloronaphthalene	1335-87-1	
Heptachloronaphthalene	32241-08-0	

^{**}Note: These CAS numbers are listed incorrectly in the PBT rule. These errors will be corrected in a future update of the PBT rule. The above table includes the correct CAS numbers.

Appendix B. Reference Materials

Certified and Standard Reference Materials Available for Laboratory Analyses

Full certificates for the following reference materials are located on the Product Testing SharePoint.

ERM®- EC680k Low Density Polyethylene		
Analysta	Mass Fraction (mg/Kg)	
Analyte	Certified Value	Uncertainty
Arsenic	4.1	0.5
Bromine	96	4
Cadmium	19.6	1.4
Chlorine	102.2	3
Chromium	20.2	1.1
Mercury	4.64	0.2
Lead	13.6	0.5
Sulfur	76	4
Antimony	10.1	1.6
	Indicative Values (mg/kg)	
Tin	15.3	2.8
Zinc	137	20
	Additional Material Information	
Acid digestible Chromium	2.9 - 16.2 mg/kg	

 $Reorder\ at:\ \underline{http://www.sigmaaldrich.com/catalog/product/sial/ermec680k?lang=en\®ion=US}\ \underline{http://www.erm-crm.org}$

CRM- PVC001 Phthalates in Polyvinyl Chloride (PVC)		
Components	CAS#	Concentration
Diisodecyl phthalate	26761-40-0	30000 μg/g
Diisononyl phthalate	28553-12-0	30000 μg/g
Bis(2-Ethylhexyl)phthalate	117-81-7	3000 μg/g
Butylbenzyl phthalate	85-68-7	3000 μg/g
Diethyl phthalate	84-66-2	3000 μg/g
Dimethyl phthalate	131-11-3	3000 μg/g
Di-n-butyl phthalate	84-74-2	3000 μg/g
Di-n-octyl phthalate	117-84-0	3000 µg/g

Reorder at: http://www.spexcertiprep.com/products/product_organic.aspx?part=CRM-PVC001

CRM-PE002 Phthalates and BPA in Medium Density Polyethylene		
Components	CAS#	Concentration
Diisodecyl phthalate	26761-40-0	30000 μg/g
Diisononyl phthalate	28553-12-0	30000 μg/g
Bis(2-Ethylhexyl)phthalate	117-81-7	3000 μg/g
Bisphenol A	80-05-7	3000 μg/g
Butylbenzyl phthalate	85-68-7	3000 µg/g
Diethyl phthalate	84-66-2	3000 μg/g
Dimethyl phthalate	131-11-3	3000 μg/g
Di-n-butyl phthalate	84-74-2	3000 μg/g
Di-n-octyl phthalate	117-84-0	3000 μg/g

Reorder at: http://www.spexcertiprep.com/products/product_organic.aspx?part=CRM-PE002

Appendix C. Glossary, Acronyms, and Abbreviations

Acronyms and Abbreviations

CSPA Children's Safe Product Act

DMA-AAS Direct Mercury Analysis Atomic Absorption Spectrophotometry

EAP Environmental Assessment Program

e.g. For example

Ecology Washington State Department of Ecology EPA U.S. Environmental Protection Agency FTIR Fourier Transform Infrared Spectroscopy

et al. And others

GC Gas Chromatography

GC-MS Gas Chromatography Mass Spectrometry

HQ Ecology Headquarters in Lacey

HPLC High Performance Liquid Chromatography

HPLC-MS High Performance Liquid Chromatography Mass Spectrometry HR-GC-MS High Resolution Gas Chromatography Mass Spectrometry

HWTR Hazardous Waste and Toxics Reduction

ICP-MS Inductively Coupled Plasma Mass Spectrometry

i.e. In other words

LC/MS/MS Liquid Chromatography-Tandem Mass Spectrometry

LCS Laboratory control sample

MEL Manchester Environmental Laboratory

MQO Measurement quality objective PBDE polybrominated diphenyl ethers

PBT persistent, bioaccumulative, and toxic substance

PCB polychlorinated biphenyls PTDB Product testing database

QA Quality assurance

QAPP Quality Assurance Project Plan

QC Quality control

RCW Revised Code of Washington

RL Reporting Limit

RPD Relative percent difference
RSD Relative standard deviation
SOP Standard operating procedure
SRM Standard reference materials

TIP Toxics in Packaging

WAC Washington Administrative Code

XRF X-ray Fluorescence

Units of Measurement

mg/Kg milligrams per kilogram (parts per million)

ppb parts per billion

QAPP: Product Testing Program – Version 1.0 Page 44 - June 2016 ppm parts per million

ug/g micrograms per gram (parts per million)

Quality Assurance Glossary

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

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*. (USGS, 1998)

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 - The difference between the population mean and the true value. Bias usually describes a systematic difference reproducible over time and is characteristic of both the measurement system and the analyte(s) being measured. Bias is a commonly used data quality indicator (DQI). (Kammin, 2010; Ecology, 2004)

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, 1997)

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, 1997)

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)

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 qualifiers - Codes used by product testing and in the product testing database.

- No qualifier, data are usable for intended purposes.
- E, the chemical was found, but there is some uncertainty about the reported amount because the instrument was not calibrated with a high enough standard.
- J, estimate, the chemical was found in the sample but there is some uncertainty about the reported amount.
- N, there is some uncertainty regarding both the identification of the chemical found, but the amount reported is accurate.
- NJ, there is some uncertainty regarding both the identification of and amount of the chemical. These results should be used with caution.
- REJ, data are unusable for all purposes. Sample results rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
- U, the analyte was not detected at or above the reported result.
- UJ, the analyte was not detected at or above the reported estimate.

Data Quality Indicators (DQI) - DQIs are 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) - DQOs are 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 - An analyte-specific and sample-specific process that extends the evaluation of data beyond data verification to determine the usability of a specific data set. It involves a

detailed examination of the data package, using both professional judgment and objective criteria, to determine whether the MQOs for precision, bias, and sensitivity have been met. It may also include an assessment of completeness, representativeness, comparability, and integrity, as these criteria relate to the usability of the data set. Ecology considers four key criteria to determine if data validation has actually occurred. These are:

- Use of raw or instrument data for evaluation
- Use of third-party assessors
- Data set is complex
- Use of EPA Functional Guidelines or equivalent for review

Examples of data types commonly validated would be:

- Gas Chromatography (GC)
- Gas Chromatography-Mass Spectrometry (GC-MS)
- Inductively Coupled Plasma (ICP)

The end result of a formal validation process is a determination of usability that assigns qualifiers to indicate usability status for every measurement result. These qualifiers include:

- No qualifier, data are usable for intended purposes
- U, the analyte was not detected at or above the reported result
- J (or a J variant), data are estimated, may be usable, may be biased high or low
- REJ, data are rejected, cannot be used for intended purposes (Kammin, 2010; Ecology, 2004)

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, 1997)

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) - 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. (USEPA, 1997)

Matrix spike - A QC sample prepared by adding a known amount of the target analyte(s) to an aliquot of a sample to check for bias 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. (EPA, 1997)

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) - This definition for detection was first formally advanced in 40CFR 136, October 26, 1984 edition. MDL is defined there as the minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, and reported to be greater than zero. (Federal Register, October 26, 1984)

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

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:

$$[Abs(a-b)/((a+b)/2)] * 100$$

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).

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)

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

Reporting limit - The lowest concentration of interest.

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, 1997)

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, 1997)

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, 1997)

Split sample - The term *split sample* denotes when a discrete sample is further 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)

References for QA Glossary

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