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Quality Assurance Project Plan

Flame Retardants in General Consumer and Children's Products

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This project is being funded by the National Estuary Program (NEP) of the U.S. Environmental Protection Agency (EPA), Region 10, Seattle, Washington. All studies conducted by the Washington State Department of Ecology (Ecology) must have an approved Quality Assurance Project Plan (QAPP). This plan describes the objectives of the study and the procedures to be followed to achieve those objectives. After completing the study, Ecology will post a report of the study to the Internet.

The plan for this study is available on the Department of Ecology's website at http://www.ecy.wa.gov/biblio/1207025.html.

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Quality Assurance Project Plan

Flame Retardants in General Consumer and Children's Products

August 2012

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HWTR-HQ: Hazardous Waste and Toxics Reduction Program
W2R: Waste 2 Resources Program

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Abstract

Polybrominated diphenyl ethers (PBDEs) are a class of persistent, bioaccumulative and toxic (PBT) compounds that were used historically as flame retardants in a wide range of consumer products. PBDEs are ubiquitous in air, soil and sediment and are building up in animals throughout the food chain. They are released from various products and transported via air and storm water to the environment. Higher levels of PBDEs are found in urban areas, making PBDE contamination particularly relevant to the highly urbanized Puget Sound Basin. PBDEs have been detected in harbor seal pups, migrating salmon and '…PBDEs are bioaccumulating to high concentrations in Puget Sound killer whales.'(Ecology, 2011d).

The Washington State Department of Ecology's (Ecology) Hazardous Waste and Toxics Reduction (HWTR) and Waste 2 Resources (W2R) Programs are conducting a study to evaluate presence of flame retardant chemicals such as PBDEs, in general consumer and children's products. Goals of the study, funded by the US Environmental Protection Agency's National Estuary Program (NEP), are to 1) determine compliance with Washington State's ban on the PBDE class of flame retardants, 2) evaluate the level of substitute flame retardants in various consumer products, and 3) verify compliance with Washington's Children's Safe Product Act (CSPA) for flame retardants on the list of Chemicals of High Concern to Children (CHCCs).

X-ray fluorescence (XRF) will be used to detect levels of bromine and chlorine which are potentially indicative of halogenated flame retardants.

Background

PBDEs and related compounds

Polybrominated diphenyl ethers (PBDEs) are a class of persistent, bioaccumulative and toxic (PBT) compounds that historically were used as flame retardants in a wide range of consumer products. PBDEs were added to plastics, upholstery fabrics and foams in such common products as computers, televisions, furniture and carpet pads. These chemicals were efficient flame retardants as well as cost effective, hence their wide use (Ecology, 2006). For deca-BDE, the largest use was in electronic enclosures, particularly in computers and televisions. PBDEs are not chemically bonded to the matrices of those materials and products. Therefore they potentially escape from their matrix through release to the air and also bind to dust.

Studies indicate that PBDEs are ubiquitous throughout the natural environment, in air, soil and sediments, and are building up in animals throughout the food chain. PBDEs were detected in migrant Chinook salmon tissue and their stomach contents from four sites in Puget Sound. Other studies have demonstrated thyroid effects on adult fathead minnows and increased risk of disease in juvenile Chinook salmon. There is evidence of bioaccumulation of PBDEs in marine mammals at high concentrations in blubber, including in Puget Sound killer whales. PBDE levels have also been observed to be increasing in orcas resident in the Puget Sound Basin. In conjunction with PBDEs concentrations, concern have been raised about the '…*increasing evidence of effects on reproductive health, the immune system, and development in exposed mammals*.' (Ross, 2006)

Total PBDE loading to Puget Sound from the major pathways assessed by the Puget Sound Toxics Loading Analysis is between 28 and 54 kilograms per year. Atmospheric deposition accounts for the largest pathway, followed by wastewater treatment plants and surface runoff. PBDEs released to air and atmospheric transport delivers them directly to Puget Sound. PBDEs deposited on land are also mobilized during storm events and delivered to surface waters. PBDEs are released in the highest quantities in commercial areas compared to other land covers, making PBDE contamination especially relevant to the highly urbanized Puget Sound Basin (Ecology, 2011c).

There are three main types of PBDEs used in consumer products: penta-BDE, octa-BDE and deca-BDE. Each has different uses and toxicity. In 2001, the total PBDE volume worldwide was estimated at over 67,000 metric tons, including 56,100 metric tons of deca-BDE. Manufacturers of penta-BDE and octa-BDE agreed voluntarily to stop producing these two forms of PBDEs at the end of 2004 (Ecology, 2006). In 2009, the US Environmental Protection Agency announced another voluntary agreement to '*...end production, importation and sales of decaBDE for most uses in the United States by December 31, 2012*' (EPA, 2010). Companies have found alternatives for most PBDE uses.

In addition to the voluntary efforts undertaken by the EPA, several states have banned the use of PBDEs in products sold within their state. In 2007, the Washington State Legislature passed legislation (Chapter 70.76 RCW) banning the use of the penta- and octa-BDE mixtures, banning the use of deca-BDE in one application (mattresses) and providing the possibility of further bans on its use if a safer alternative could be identified (Washington, 2007). Ecology and the Washington State Department of Health (DOH) conducted an alternatives assessment to determine if safer alternatives to deca-BDE exist for computers, televisions and residential upholstered furniture. As a result of this assessment, a safer alternative, resorcinol diphenyl phosphate (RDP), was identified in 2009. As of January 1, 2011, deca-BDE is banned in all three applications. (Ecology, 2009).

Deca-BDE was also identified by Ecology as a chemical of high concern to children (CHCC). In 2008, the Washington State Legislature passed legislation (Chapter 70.240 RCW) that requires Ecology to identify chemicals that are both toxic and have a potential exposure pathway to children (Washington, 2008). As directed by this legislation, Ecology published a list of 66 CHCCs and deca-BDE was one of the 66 (Ecology, 2011b). Deca-BDE was placed on this list because of its carcinogenic potential, the tendency of deca-BDE to degrade to more toxic forms of PBDEs such as found in the penta- and octa-BDE mixtures and children's potential exposure to deca-BDE from it's wide use as a flame retardant (DOH, 2010). Any manufacturer that uses deca-BDE in children's products manufactured or sold in Washington State, must report to Ecology (Ecology, 2011a). A list of CHCCs can be found in Appendix A.

With the banning of PBDEs, manufacturers have produced a number of alternatives. One alternative that has garnered considerable attention are the polybrominated diphenyl ethanes (PBDPEs). These chemicals are similar to PBDEs. No toxicity data, however, was required to allow this chemical onto the market and concerns have been raised that similarities between PBDPEs and PBDEs might harbinger similar toxicological impacts.

Chlorinated phosphate flame retardants

In addition to deca-BDE mentioned above, a chlorinated phosphate flame retardant, tris(2-chloroethyl) phosphate (TCEP), was also identified as a CHCC (Appendix A). Recently, a similar compound, tris(1,3-dichloro-2-propyl) phosphate (TDCPP), was identified by the California Environmental Protection Agency as a chemical known to cause cancer and placed upon the Proposition 65 list of toxic chemicals (Cal EPA, 2011).

A third flame retardant compound in this class, tris(1-chloro-2-propyl) phosphate (TCPP) has also generated some concern. The Organization of Economic and Cooperative Development (OECD) has indicated that TCPP is harmful to aquatic organisms (OECD, 2012). Given the similarity of this compound, however, to TCEP and TDCPP concerns have arisen about its widespread use.

Chlorinated phosphates including TCEP have been found in a number of environmental media including effluent from sewage treatment plants, precipitation (rain, snow and glacial ice) and surface waters, primarily in Europe (EU, 2009). TCEP has also been found in dust in homes, schools, hospitals and various other locations (EC, 2009).

TCEP and TCPP are slightly toxic to aquatic organisms at all trophic levels and TDCPP is moderately toxic to fish. All three compounds are slightly toxic to terrestrial species, aquatic green algae and are non-toxic to sewage bacteria (NICNAS, 2001). Organophosphates including TCEP and TDCP have also been found in fish (perch and carp) in a limited study in Sweden (Marklund, 2005).

Non-halogenated phosphate flame retardants

As mentioned previously, Ecology identified RDP as a safer alternative to deca-BDE, especially in the computer and television industry (Ecology, 2009). Other non-halogenated flame retardants were also reviewed in the same report. One of the flame retardants evaluated as an alternative, triphenyl phosphate (TPP), was eliminated due to its high environmental toxicity (Ecology, 2009). Data is lacking on whether these chemicals are being widely used and if concerns about TPP warrant further evaluation.

In one study in Belgium, TPP was found in all of the 33 dust samples tested (Van den Eede, 2011). In a U. S. study, TPP was found in 98% of the 50 house dust samples tested (Stapleton, 2009). TPP has also been found in environmental samples. TPP was found in fish in a Swedish study of lakes and coastal regions (Sundkvist, 2010). TPP was detected in almost half of the fish sampled from Manila Bay in the Philippines (Kim, 2011). It was found in reclaimed wastewater (Loraine, 2006) and was detected in blubber of six bottlenose dolphins from the Gulf of Mexico (Kuehl, 1995). Suckling dolphins had levels nearly 10 times greater than adult males.

Flame retardants in products

Recent studies have shown that many of the above flame retardants can still be found in products. Stapleton et al. studied polyurethane foam used in baby products and identified that all three chlorinated phosphate flame retardants were found in a wide range of products including car seats, changing table pads, sleep positioners, portable mattresses, nursing pillows, baby carriers, high chairs and infant bath mat/slings (Stapleton, 2011). One of the non-halogenated flame retardants, TPP, was found in a number of similar products. Penta-BDE was found in some of the products but the authors cautioned these were older products potentially manufactured before specific bans were implemented.

In an earlier study, chlorinated phosphate flame retardants were found in a number of products ranging from chairs, futons and ottomans to more child-oriented products like a nursery glider/rocker and baby stroller (Stapleton, 2009).

The concentration of flame retardants in house dust is widely used to monitor the presence of flame retardants in consumer products. Because many household products contain flame retardants that can be released and accumulated in house dust, dust has become a common sampling media. This connection has been investigated and shown to have a high correlation between the amount and type of consumer products and the amount of PBDEs found in house dust (Allen, 2008). Alternative and new brominated flame retardants like decabromodiphenyl ethane (deca-BDPE) were detected in house dust indicating that use of this alternative flame retardant is increasing (Stapleton, 2008).

During creation of the CHCC list, Ecology reviewed several sources of data on chemicals in products. Two sources that proved particularly useful were studies conducted by the Danish Environmental Protection Agency and similar bodies of the Dutch Government. In both instances, the organizations purchased products and evaluated them for a wide range of chemicals. This information was used to identify potential sources of exposure to children and many of the flame retardants in this study were found in general consumer and children's products. For example, TCEP was found in electronics, perfume in toys and children's articles (authors stated it is more likely to be present as a flame retardant than part of the perfume itself), and plastic toys. TPP was found in electronics, baby products and plastic toys. Penta-, octa- and deca-BDE were all found in electronics while deca-BDE was also found in indoor air sampling from various consumer products (Stone, 2010).

The objective of this study is to identify products that are not in compliance with the Washington legislation banning the use of PBDEs. Included in this evaluation are two phosphate flame retardants that were identified as possible alternatives to deca-BDE in specific applications. In addition, products containing flame retardants identified as CHCCs will be tested to evaluate compliance with CSPA reporting requirements.

Project Description

Ecology's HWTR and W2R Programs will conduct a study that uses a portable XRF instrument to screen for concentrations of bromine and chlorine[1] indicate the possible presence of certain flame retardants (Table 1) in various consumer products. The objectives of the study are to:

- Determine compliance with the Washington State ban on the PBDE class of flame retardants.
- Assess the levels of flame retardants in general consumer and children's products.
- Determine compliance with the state's CSPA reporting requirements for flame retardants which also appear on the CHCC list.

Products will be purchased and screened for bromine and chlorine with a portable XRF analyzer during the summer of 2012. Those samples found to contain bromine and chlorine using the XRF will be further evaluated to determine if they contain any of the flame retardants of interest. Special emphasis will be placed on whether chlorine detected by XRF is possibly due to chlorinated flame retardants or plastics like polyvinyl chloride. An XRF cannot differentiate between the two potential sources of chlorine.

Information from a number of sources will be searched to identify potential products containing these compounds. Sources will include but are not restricted to the results of peer-reviewed scientific studies, databases compiling product information (National Institute of Health's Household Product Database, Environmental Working Group's Skin Deep Database, etc.) and sampling reports from authoritative bodies (Danish Environmental Protection Agency, Dutch

[1] The ASTM method does not include the analysis of chlorine; however, the XRF manufacturer has indicated that chorine can be detected with an XRF at concentrations well below the method detection limit.

Government, etc.). Samples possibly containing any of the flame retardants of interest will be sent to a contract laboratory for analysis.

Sampling Process Design (Experimental Design)

Approximately 300 products will be gathered for testing during the sampling event. Information will be assimilated from a number of different sources to identify products that may potentially contain some of the flame retardants of interest.

All product samples will be screened with a portable XRF for bromine and chlorine to determine if the products may contain any of the flame retardants of interest. As an XRF cannot detect the actual presence of the specific flame retardants, information on the label, the type of plastic used and other potential sources of information will be used to determine whether a product sample is likely to contain any of the flame retardants of interest. Those products that contain bromine and chlorine **and** could potentially contain a flame retardant of interest will be given highest priority and sent to a laboratory and analyzed using EPA approved laboratory methodologies to determine if any of the flame retardants of interest are present and, if so, at what concentration. If insuffienct samples are found that meet these criteria, samples that are found to contain chlorine or bromine but for which there is no documented evidence that flame retardants are likely to be present will be sent for analysis. It is anticipated that approximately 175 product samples will be forwarded for laboratory for analysis.

Product Selection

Screening during the sampling event will focus on products that historically have contained banned flame retardants and products likely to contain the flame retardants of interest based upon information gleaned from other resources and products and product components that are most likely to impact the Puget Sound. Penta- and Octa-BDE are banned for all uses with the exception of some products specifically identified in the legislation. The deca-BDE ban specifically covers mattresses, computers, televisions and residential upholstered furniture. In addition, the ban on penta- and octa-BDE covers all but a few uses specifically called out in the legislation. Products that historically contained penta- and octa-BDE will be sampled to assess whether manufacturers are using PBDE alternatives. Samples from these sources will be collected to verify compliance with Ecology's PBDE ban.

Sample selection will also focus on products or product components likely to be mouthed or used by children under three. Under the CSPA Reporting Rule tiered approach, Tier 1 products are those intended to be put into a child's mouth, applied to their skin, or any mouthable product for a child less than 3. Tier 1 products must be reported first. Tiers 2 – 4 include products intended for prolonged direct skin contact, short-duration direct skin contact, and no intended skin contact, respectively. Product analysis will emphasize Tier 1 products and products likely to contain flame retardants of interest unless sufficient samples cannot be obtained.

Products will be selected that meet the following criteria:

1. Products that previously contained PBDEs prior to the ban.

2. Products that come in contact with children under 3 years old and are likely to contain flame retardants of interest.
3. Products that may be mouthed by children under 3 years old that may contain flame retardants.

Products will be selected based upon several sources including but not limited to:

- Ecology research such as the PBDE Chemical Action Plan and the deca-BDE Alternatives Assessment that identified products historically containing PBDEs.
- Peer-reviewed scientific articles that have tested products containing PBDEs or other flame retardants of interest found in childrens products. For example, the study of children's products conducted by Stapleton et al. (2011) and similar studies will be used to identify products of interest.
- Product databases and other information available on the internet that indicates the possible presence of flame retardants in products that meet the above criteria.

Products that will be considered for possible analysis include but are not limited to:

Products	**Justification**
Electronic casings	Historically used deca-BDE[+]
Upholstered furniture	Historically used deca-BDE[+]
Foam cushions	Historically used penta-BDE[++]
Foam baby products	Historically used penta-BDE[+++]
Car Seats	Found to contain TCEP or TDCPP[+++]
Nursing Pillows	Found to contain TCEP or TDCPP[+++]
Sleep positioners	Found to contain TCEP or TDCPP[+++]
Nursing pillows	Found to contain TCEP or TDCPP[+++]

[+] See Ecology and DOH deca-BDE Alternatives Assessment for more details (Ecology, 2009)
[++] See Ecology and DOH PBDE Chemical Action Plan for more details (Ecology, 2008)
[+++] See Stapleton et al (2011) for more details.

Product Screening

Products will be screened using a portable XRF analyzer according to the following:

- Manufacturer's standard operating procedures (Niton, 2011).
- Ecology recommendations (Ecology, 2012b).
- Approved and validated methods such as ASTM method F 2617-08 *Standard Test Method for Identification and Quantification of Chromium, Bromide, Cadmium, Mercury, and Lead in Polymeric Material Using Energy Dispersive X-ray Spectrometry (ASTM, 2008).*

Target Chemicals and Screening Criteria

Target chemicals proposed for testing, and recommended practical quantitation limits (PQLs) for laboratory analysis of each, are shown in Table 1.

Table 1. Analytes of Interest

Analytes	CAS Number	PQL (ppm+++)
PBDEs		
Pentabromodiphenyl ether mix	32534-81-9	100+
Octabromodiphenyl ether mix	32536-52-0	100+
Decabromodiphenyl ether	1163-19-5	100+
Decabromodiphenyl ethane	84852-53-9	100+
Chlorinated phosphates		
TCEP	115-96-8	100++
TCPP	13674-84-5	100++
TDCPP	13674-87-8	100++
Non-haloginated phosphates		
RDP	115-86-6	100++
TPP	125997-21-9	100++

+ The PQL for this study is set at 10% of the de minimis level of 1,000 ppm for compliance with the Washington ban on PBDE flame retardants (Ecology, 2008).

++ No PQL exists for these compounds; the PQL for PBDEs is used as a surrogate.

+++ ppm = parts per million of flame retardant by weight.

Flame retardants are commonly used in products at percent levels (Stapleton et al., 2011), which is two orders of magnitude above the PQLs. Samples containing the highest levels of flame retardants of interest (based on screening results for bromine and chlorine and all other available information such as labels, product databases and other readily-available sources) will be sent to the laboratory for analysis. The exact number of samples will depend upon the availability of applicable products and budgetary constraints.

Organization and Schedule

Table 2 lists the individuals involved in the project and Table 3 contains a schedule.

Table 2. Organization of Project Staff and Responsibilities

Staff	Title	Responsibilities
Joshua Grice, W2R (360) 407-6786	Client	Clarifies scopes of the project. Provides internal review of the QAPP and approves final QAPP.
Alex Stone HWTR-HQ Program (360) 407-6758	Project Manager	Writes QAPP, oversees field sampling and transportation of samples to laboratory. Conducts QA review of data, analyzes and interprets data. Writes draft report and final report.
Ken Zarker, HWTR-HQ (360) 407-6698	Manager for Project Manager	Reviews project scope and budget, tracks progress, reviews draft QAPP and approves final QAPP.
Tom Gries (360) 407-6327	NEP QA Coordinator	Reviews draft QAPP and recommends approval and reviews draft report.
William R. Kammin, EAP (360) 407-6964	Ecology QA Officer	Reviews draft QAPP and approves final QAPP.
Carol Kraege, W2R (360) 407-6906	Client	Reviews project scope and budget, tracks progress, reviews the draft QAPP and approves final QAPP and expenditure of funds for implementation of the QAPP.

HWTR-HQ: Hazardous Waste and Toxics Reduction Program-Headquarters.
QAPP: Quality Assurance Project Plan.
W2R: Waste 2 Resources.

Table 3. Proposed Schedule for Completing Field and Laboratory Work and Reports

Field and laboratory work	Due date	Lead staff
Field work completed	September 2012	Alex Stone
Laboratory analyses completed	December 2012	
Final report		
Author lead / Support staff	Alex Stone	
Schedule		
Draft due to supervisor	February 2013	
Draft due to client/peer reviewer	April 2013	
Final (all reviews done)	May 2013	
Final report due on web	June 2013	

Sample Collection and Preparation

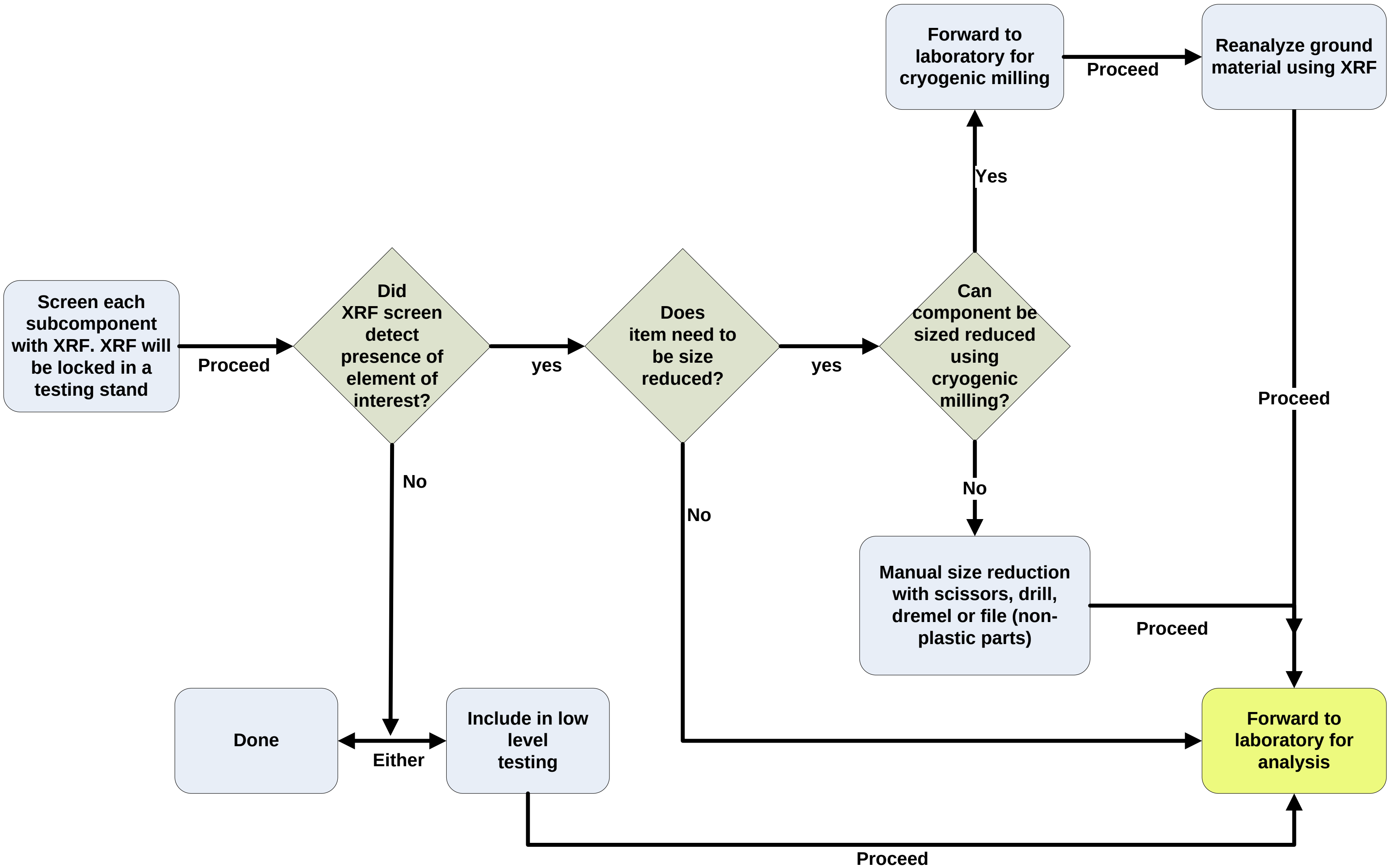
Products will be obtained in person or through internet retailers by HWTR or W2R staff. In addition, products reserved from other Ecology sampling events (Ecology, 2012) will be evaluated to determine if they meet the requirements of this QAPP. Samples will be prepared using the standard operating procedure developed for this sampling event (Ecology, 2012a). A brief summary of this procedure is described below.

Upon collection, products will be removed from their original packaging using pre-cleaned stainless steel implements. Products will be separated into to three fractions. Fraction 1 will consist of the product packaging that will be retained for possible analysis under a separate QAPP. Fraction 2 will comprise the package contents or product in its entirety. Fraction 3 will consist of the container used to hold the product ingredients. If necessary, Fraction 3 may be further broken down into individual components. Items with different colors or base materials will be treated as components. Additionally, individual pieces of products intended to be disassembled will be treated as components.

For example, children's modeling clay might be purchased and tested. Toys like this come packaged in a combination of clear plastic and paper or cardboard. The modeling clay itself is inside a plastic container. For the purposes of this study, the toy would be separated into three fractions. The packaging would be the external plastic and paper or cardboard (fraction 1), the plastic container holding the modeling clay (fraction 3) and the modeling clay itself (fraction 2). Although modeling clay may not be tested for flame retardants, similar decisions may be necessary on a case-by-case basis depending upon the product and how it is packaged and presented.

Components targeted for testing will be removed with stainless steel tools (scissors, pliers, saws, etc.) for further testing. All tools will be cleaned following the sequence identified above. Some samples such as those consisting of hard plastic or other unique construction may be sent out for cryomilling to facilitate release of the chemicals of interest from the plastic matrix during extraction and sample preparation. Cryomilling refers to the process of reducing a sample to very small particle sizes by employing cryogenic temperatures and a mechanical mill. Milled samples will be screened by XRF in the stand. Cryomilling decisions will be made on a case-by-case basis. Non-plastic items such as foams, textiles and metals will be reduced in size using a file, drill, dremel tool or scissors. Scrapings will be further ground (if the material allows) by mortar and pestle. Sub-sampled materials (ground, cut or scraped) will be reanalyzed prior to laboratory testing. Ecology will identify a company who can cryomill these samples. Figure 1 presents a schematic of the screening and preparation procedure.

Figure 1. Screening and Sample Preparation Schematic



Product samples will be sent to Ecology's Manchester Environmental Laboratory (MEL) or a contract laboratory for organic analysis. The procedure to be followed to identify a laborabory for sample analysis is as follows:

1. Manchester will be approached first to run the samples.
2. If Manchester declines, laboratories under general contract to Washington State to provide analytical support (State Contract 1807[2]) will be approached.
3. If no general contract laboratory can conduct the analyses for any reason (not accredited, sample preparation concerns, etc.), the Project Manager will solicit qualified and accredited laboratories to provide analytical services using established Ecology contracting procedures. The Project Manager will be responsible for the review and evaluation of all potential contract laboratories to guarantee the laboratory meets the requirements of this Plan.

Photos and descriptive notes on each product screened such as approximate thickness, surface roughness, material makeup, etc. will be recorded. Other information such as the type of advertisement used to sell the product, where in the store the product was located, etc. may be collected for the purpose of CSPA compliance.

All field and laboratory staff handling the items will wear powder free nitrile gloves. Stainless steel tools used to deconstruct the product or remove it from its products along with the mortar and pestle will be cleaned by the following sequence: hot water scrub with liquinox soap, 10% nitric acid rinse and three rinses with deionized water.

Analytical Procedures

XRF Analysis

Figure 2. Niton Portable XRF

Individual product components will be screened at Ecology using a Niton XL3t portable XRF analyzer (Figure 2) following standard operating procedures created by the manufacturer (Niton, 2011) and ASTM method F 2617-08 *Standard Test Method for Identification and Quantification of Chromium, Bromide, Cadmium, Mercury, and Lead in Polymeric Material Using Energy Dispersive X-ray Spectrometry*. In addition, EPA has published Method 6200, *Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment* (EPA, 2007). Although Method 6200 is for soils and sediments, it does demonstrate

[2] Washington State Contract 1807 for Analytical Laboratory Services can be found at: https://fortress.wa.gov/ga/apps/ContractSearch/ContractSummary.aspx?c=01807.

that the XRF can also be used for non-polymeric materials which is in agreement with the manufacturer's specifications (Niton, 2011). No testing will be done in the field.

For the initial screening, a reading will be taken for at least 30 seconds on a smooth (or near smooth) area of the packaging large enough to cover the spectrometer's window and at least 2 mm thick. If the item is less than 2 mm thick, it may be folded on to itself until 2 mm depth has been reached (care will be taken to trap minimal air in between folds).

If the screening measurement violates screening criteria, a second longer measurement will be taken (up to 120 seconds). Both measurements will be taken using the appropriate XRF software package (based on sample material). Limits of Quantification (LOQs) as identified by the manufacturer are shown in Table 4. After XRF analyses are completed, samples will be placed in pre-cleaned I-Chem jars and forwarded to the appropriate laboratory for testing.

Table 4. Niton Portable XRF LOQs and Expected Range of Results

Element	Expected Range of Results (ppm)	LOQ (ppm)$^{+}$
Chlorine	<LOQ - 300	15
Bromine	<LOQ - 300	15

ppm = parts per million
LOQ = Limit of Quantification
$^{+}$ Polyethylene blank, 8 mm aperture, 120 second total analysis time

All samples screened will be assigned a unique identifier and results from the XRF will be transferred to Microsoft Excel spreadsheets.

Laboratory

Standard methods will be used to measure extractable concentrations of flame retardant compounds. These methods, along with estimated reporting lmit (RLs), are listed in Table 5. At a minimum, sample extraction shall consist of EPA Solid-Waste 846 Method 3540 (EPA, 1996).

Extracts will be analyzed using a gas chromatograph/mass spectrograph (GC/MS) using Solid Waste-846 method 8270 (EPA, 1996).

Table 5. Laboratory Methods and Reporting Limits

Analyte	Digestion Method	Instrumentation	Method	RL^{++} (ppm)$^{+++}$
PBDEs	3540	GC/MS^{+}	EPA 8270	10.0
Decabromodiphenyl ethane	3540	GC/MS^{+}	EPA 8270	10.0
Chlorinated phosphates	3540	GC/MS^{+}	EPA 8270	50.0
Non-halogenated phosphates	3540	GC/MS^{+}	EPA 8270	30.0

+GC/MS = Gas chromatography/mass spectroscopy
++RL = Reporting Limit
+++ppm = parts per million of analyte in sample by weight

Budget

The project budget is included in Table 6.

Table 6. Project Budget

	# of Samples	Cost per sample	Total
Products purchased	300	$30.00	$9,000.00
Flame retardants	175	$1,000.00	$175,000.00
Total			**$184,000.00**

Quality Objectives

Quality objectives for this project are to obtain data of sufficient quality so that the amount of flame retardant compounds in a representative subsample of general consumer and children's products can be determined. These objectives will be achieved through careful attention to the sampling, sample processing, measurement, and quality control (QC) procedures described in this plan.

Measurement Quality Objectives

An XRF reading will be taken on standards provided by the manufacturer at a 5% frequency (one every 20 samples). Measurement quality objectives (MQOs) are based upon the results of a previous Ecology study on the use of XRF as a screening tool (Ecology, 2012b). The XRF analysis will be performed per the manufacturer's specification (Niton, 2011). Plastic and metal standards are provided by the manufacturer to meet exact specifications and will be used to test whether the XRF is operating to manufacturer's standards. XRF results will be compared with these standard results to verify validity of testing results.

Performance of the portable XRF has been determined in a previous EAP report (Ecology, 2012), which demonstrated that the XRF can be used effectively as a screening tool, particularly for metals. The conclusions from the report will be implemented in this work where possible and, as recommended in this study, all screening will be done using a stand to minimize error.

MQOs for analysis of elements and flame retardant compounds are shown in Tables 6 and 7, respectively. For the XRF analyses, these criteria are required during screening analyses conducted by Ecology staff. For the laboratory analyses, it is expected that MEL and contract laboratories will meet these criteria. MQOs falling outside of the acceptance limits will be reviewed by the Project Manager for their usability.

Table 7. MQOs for XRF Analyses

Analyte	**Manufacturer's standards** (ppm)	**Duplicates**[+] (RPD)[++]	**Blanks** (ppm)[+++]
Bromine and chlorine[+]	± 25%	± 25%	< 1.0

+ Chlorine was not a target compound in the Ecology study (Ecology, 2012b). Similar MQOs, however, are established for chlorine based upon the bromine results.

Table 8. MQOs for Laboratory Analyses

Analyte	**Laboratory Control Samples** (recovery)	**Matrix**[+] **Spikes** (recovery)	**Duplicates**[+] (RPD)[++]	**Method Blanks** (ppm)[+++]	**Surrogate Recovery** (recovery)
PBDEs	85 - 115%	40-140%	± 25%	< 1.0	30-150%
PBDPE	85 - 115%	40-140%	± 25%	< 1.0	30-150%
Chlorinated phosphates	85 - 115%	40-140%	± 25%	< 1.0	30-150%
Non-halogenated phosphates	85 - 115%	40-140%	± 25%	< 30.0	30-150%

[+] Matrix spike duplicates and split duplicates
[++]RPD = Relative Percent Difference
[+++]ppm = parts per million

Quality Control Procedures

Screening

No sampling will be conducted in the field. All samples will be purchased and brought back to Ecology for screening; therefore, no field quality control procedures are anticipated for this project. Real-time results will be compared to MQOs through the use of standards, duplicates and blanks as identified in Table 7. Any corrective actions to account for problems such as instrument drift will be documented in the final report. If warranted, these issues will be noted and corrective actions taken such as recalibration and re-running of samples.

Laboratory

Table 9 displays the laboratory QC tests planned for flame retardant analysis. Laboratory QC tests will consist of laboratory control samples, matrix spikes, matrix spike duplicates, laboratory duplicates, and method blanks. Final flame retardant results will be corrected for surrogate recovery. The laboratory will test one methanol blank in addition addition to the method blank per batch of 20 samples processed.

Table 9. Quality Control Tests

LCS	Matrix Spkes	Matrix spike Duplicates	Laboratory Duplicates	Method Blanks	Surrogate Recover
1/batch	1/batch	1/batch	1/batch	1/batch	Every sample

LCS: Laboratory Control Sample
Batch: maximum of 20 samples

Data Management Procedures

XRF data from the screening portion of the project will be transferred to Microsoft Excel spreadsheets and managed by the Project Manager. All data will be stored on the Reducting Toxics Threat Sharepoint site, which is copied to storage every night to maintain data in case of hardware or other unexpected problems. In addition, a database is under development that will be used to store the raw analysis data. This database will also be stored on an Ecology server and backed up daily for data protection.

Data packages from MEL and any contract lab will include case narratives discussing any problems encountered with the analyses, corrective actions taken, changes to the referenced method, and an explanation of data qualifiers. The narrative will also address condition of the samples on receipt, sample preparation, methods of analysis, instrument calibration, recovery data, and results on QC samples. This information is needed to evaluate the accuracy of the data and to determine whether the MQOs were met.

Data Verification

The Project Manager will review the data packages from the contract laboratory. Data packages will include Tier III deliverables such as calibration reports, chromatograms, and spectra benchsheets. This review will verify that (1) methods and protocols specified in this project plan were followed, (2) all calibrations, QC checks, and intermediate calculations were performed for all samples, and (3) that the data are consistent, correct, and complete, with no errors or omissions. Evaluation criteria will include the acceptability of procedural blanks, calibration, matrix spike recoveries, labeled compound and internal standard recoveries, ion abundance ratios, duplicates, laboratory control samples, and appropriateness of data qualifiers assigned.

The Project Manager will provide case narratives describing any problems encountered with the analysis, corrective actions taken, deviations from the referenced method, and an explanation of data qualifiers. The narrative should address condition of the samples upon receipt, sample preparation, methods of analysis, instrument calibration, recovery data, and results of quality control samples. This information is needed to evaluate the accuracy of the data and to determine whether the MQOs were met.

Data Quality (Usability) Assessment

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 contract laboratory met MQOs for method blanks, LCS, duplicate, and matrix spike samples. Reporting limits will be examined to ensure that the contract-defined reporting limit was met. Data will either be accepted, accepted with additional qualification, or rejected and re-analysis considered. Data quality and usability will be discussed in the final report. Data will be evaluated for false negatives or positives for any inpact they may have upon the results of the study. This includes possible impacts on enforcement or any other use of the data.

Audits

MEL and any contracted laboratory will obtain accreditation by the State of Washington for analysis of the target flame retardants by Method 8270. As part of the accreditation process, the State of Washington will perform on-site audits of the laboratory's staff, facilities, and analytical capabilities. The laboratory's quality system, test methods, records, and reports will also be evaluated as part of the accreditation process. The laboratory selected for analysis must participate in performance and system audits of their routine procedures. Results of these audits must be made available on request.

Report

A final report detailing the findings of the study will be completed. The final report will include:

- Categorical descriptions of the products screened with the XRF (some information such as brands, product names, etc. will not be included).
- Any deviations from the QAPP in terms of sample preparation, QA/QC requirements, etc.
- Comparison of laboratory results with XRF screenings, where applicable.
- Assessment of product test results from general consumer and children's products for flame retardants.
- Determination of what levels of specific flame retardants are found in general consumer and children's products.
- Data on specific products and product components and whether the levels of PBDEs found violate Washington State legislation.
- Data on specific products and product components and whether the levels of flame retardants found would require reporting as dictated by the CSPA legislation.Appendices that include final SOP and tables showing results of screening and laboratory analyses.

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Appendix A. Chemicals of High Concern to Children

CAS	Chemical
50-00-0	Formaldehyde
62-53-3	Aniline
62-75-9	N-Nitrosodimethylamine
71-36-3	n-Butanol
71-43-2	Benzene
75-01-4	Vinyl chloride
75-07-0	Acetaldehyde
75-09-2	Methylene chloride
75-15-0	Carbon disulfide
78-93-3	Methyl ethyl ketone
79-34-5	1,1,2,2-Tetrachloroethane
79-94-7	Tetrabromobisphenol A
80-05-7	Bisphenol A
84-66-2	Diethyl phthalate
84-74-2	Dibutyl phthalate (DBP)
84-75-3	Di-n-Hexyl Phthalate
85-44-9	Phthalic Anhydride
85-68-7	Butyl Benzyl phthalate (BBP)
86-30-6	N-Nitrosodiphenylamine
87-68-3	Hexachlorobutadiene
94-13-3	Propyl phthalate
94-26-8	Butyl phthalate
95-53-4	2-Aminotoluene
95-80-7	2,4-Diaminotoluene
99-76-3	Methyl phthalate
99-96-7	p-Hydroxybenzoic acid
100-41-4	Ethylbenzene
100-42-5	Styrene
104-40-5	4-Nonylphenol; 4-NP and its isomer mixtures including CAS 84852-15-3 and CAS 25154-52-3
106-47-8	para-Chloroaniline
107-13-1	Acrylonitrile
107-21-1	Ethylene glycol
108-88-3	Toluene
108-95-2	Phenol
109-86-4	2-Methoxyethanol
110-80-5	Ethylene glycol monoethyl ester

CAS	Chemical
115-96-8	Tris(2-chloroethyl) phosphate (TCPP)
117-81-7	Di-2-ethylhexyl phthalate (DEHP)
117-84-0	di-n-octyl phthalate (DnOP)
118-74-1	Hexachlorobenzene
119-93-7	3,3'-Dimethylbenzidine and Dyes Metabolized to 3,3'-Dimethylbenzidine
120-47-8	Ethyl phthalate
123-91-1	1,4-Dioxane
127-18-4	Perchloroethylene
131-55-5	Benzophenone-2 (Bp-2); 2,2',4,4'-Tetrahydroxybenzophenone
140-66-9	4-tert-Octylphenol; 1,1,3,3-Tetramethyl-4-butylphenol
140-67-0	Estragole
149-57-5	2-Ethylhexanoic Acid
556-67-2	Octamethylcyclotetrasiloxane
608-93-5	Benzene, pentachloro
842-07-9	C.I. Solvent Yellow 14
872-50-4	N-Methylpyrrolidone
1163-19-5	2,2',3,3',4,4',5,5',6,6'-Decabromodiphenyl ether; BDE-209
1763-23-1	Perfluorooctanyl sulphonic acid and its salts; PFOS
1806-26-4	Phenol, 4-octyl-
5466-77-3	2-Ethyl-hexyl-4-methoxycinnamate
7439-97-6	Mercury & mercury compounds including methyl mercury (22967-92-6)
7439-98-7	Molybdenum & molybdenum compounds
7440-36-0	Antimony & Antimony compounds
7440-38-2	Arsenic & Arsenic compounds including arsenic trioxide (1327-53-3) & dimethyl arsenic (75-60-5)
7440-43-9	Cadmium & cadmium compounds
7440-48-4	Cobalt & cobalt compounds
25013-16-5	Butylated hydroxyanisole; BHA
25154-52-3	Nonylphenol
25637-99-4	Hexabromocyclododecane
26761-40-0	Diisodecyl phthalate (DIDP)
28553-12-0	Diisononyl phthalate (DINP)

Appendix B. Glossary, Acronyms, and Abbreviations

Acronyms and Abbreviations

Following are acronyms and abbreviations used frequently in this report.

ASTM	American Society for the Testing of Materials
CHCC	Chemicals of High Concern to Children
CPSA	Children's Safe Product Act
Deca-BDE	Decabromodiphenyl ether
Deca-BDPE	Decabromodiphenyl ethane
DOH	Washington State Department of Health
EAP	Environmental Assessment Program
EC	Environment Canada
Ecology	Washington State Department of Ecology
EPA	United States Environmental Protection Agency
et al.	Et alia or and others
EU	European Union
GC-MS	Gas Chromatography-Mass Spectroscopy
HCl	Hydrochloric acid
HF	Hydrofluoric acid
HNO_3	Nitric acid
HQ	Headquarters
HWTR	Hazardous Waste and Toxics Reduction Program
i. e.	Id est or In other wordsLCS Laboratory control sample
LOQ	Limit of Quantitation
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
NEP	National Estuary Program
NICNAS	National Industrial Chemicals Noticiation and Assessment Scheme
Octa-BDE	Octabromodiphenyl ether
OECD	Organisation for Economic Cooperation and Development
PBDE	Polybrominated diphenyl ether class of flame retardants
PBDPE	Polybrminated diphenyl ethane class of flame retardants
PBT	persistent, bioaccumulative, and toxic substance
Penta-BDE	Pentabromodiphenyl ether
PPM	Parts per million
PQL	Practical quantitation limit
RCW	Revised Code of Washington
RDP	Resorcinol diphenyl phosphate
RL	Reporting limit
QA	Quality assurance
QC	Quality control
QAPP	Quality Assurance Project Plan
RPD	Relative percent difference
RSD	Relative standard deviation

SOP	Standard operating procedures
SRM	Standard reference materials
TCEP	tris (2-chloroethyl) phosphate
TCPP	tris (1-chloro-2-propyl) phosphate
TPP	Triphenyl phosphate
TDCPP	tris(1,3-dichloro-2-propyl) phosphate
W2R	Waste 2 Resources Program
XRF	X-Ray Fluorescence

Units of Measurement

ng	nanogram, a unit of mass equal to one millionth of a gram
mg	milligram, one thousandth of a gram
g	gram, a unit of mass
kg	kilograms, a unit of mass equal to 1,000 grams.
meter	meter, a unit of distance
mm	millimeter, a unit of distance equal to one thousandth of a meter
Liter	liter, a unit of volume
mL	milliliter, equal to one thousandth of a liter
ppm	parts per million
mg/kg	milligrams per kilogram (parts per million)
ng/g	nanograms per gram (parts per billion)
ng/kg	nanograms per kilogram (parts per trillion)
mg/L	milligrams per Liter (parts per million)
ng/L	nanograms per Liter (parts per trillion)
s.u.	standard units