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State of Washington

Quality Assurance Project Plan

Polychlorinated Biphenyls (PCBs) in General Consumer Products

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The plan for this study is available on Ecology's website at:
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April 2013

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Abstract

Polychlorinated biphenyls (PCBs) are a class of of manmade chlorinated organic chemicals known for their low flammability, low reactivity, low water solubility and persistence in the environment (Erickson, 1997). PCB use was restricted in 1976 by the passage of the federal legislation, the Toxic Substances Control Act (TSCA). TSCA bans certain uses and restricts PCB concentrations to less than 50 ppm (EPA, 1976). PCBs are still found in commercial products where they are intentionally used below this level or where they occur as an inadvertent contaminant in many other products. Ecology has identified three major sources of PCBs impacting the Puget Sound (Ecology, 2011d):

1. Electrical equipment spills and leakage.
2. Residential trash burning.
3. Building sealant (caulk) volatilization and abrasion.

As PBTs, PCBs are ubiquitous in air, soil and sediment and are commonly found in animals throughout the food chain. They are released from various products including pre-ban transformers still in use and transported via air and storm water to the environment. Higher levels of PCBs are found in urban areas, making PCB contamination particularly relevant to the highly urbanized Puget Sound Basin. Ecology identified PCBs as one of 7 chemicals or classes of chemicals that '… *result in documented or potentially adverse effects to a variety of aquatic organisms*.' (Ecology, 2011d).

The Washington State Department of Ecology's (Ecology) Hazardous Waste and Toxics Reduction (HWTR) Program is conducting a study to evaluate presence of PCBs in general consumer products. Particular emphasis is placed on either products known to contain PCBs as a contaminant formed during the manufacturing of dyes and pigments contained within the product or caulks known to still contain and historically have contained high levels of PCBs. Products known to contain PCBs as contaminants include paints (Wu, 2012) and newspapers, glossy magazines, cereal boxes, yellow plastic bags, etc. (Rodenburg, 2012).

All PCBs are of interest and four specific PCBs are targeted in this study:

PCB	CAS
PCBs[1]	1336-36-3
3,3'-Dichlorobiphenyl; PCB-11	2050-67-1
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl; PCB-206	40186-72-9
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl; PCB-208	52663-77-1
Decachlorodiphenylether; PCB-209	2051-24-3

[1] PCBs in the caulk samples are equivalent to the sum of the Aroclor mixtures and individual PCB congeners identified in EPA SW-846 method 8082A.

These 4 PCB congeners are not part of historical Aroclor mixtures and are believed to be byproducts from the production of dyes and pigments. The goals of the study are to 1) evaluate the level of PCBs in various consumer products including both specific congeners from known sources and identification of other PCBs from unknown sources, and 2) confirm the continued presence and potential non-point release of PCBs within Washington State. PCB contamination from known contaminated sites is a well documented point source for PCBs in Washington state; PCBs from new sources including those included in this study may indicate an additional source of PCBs released to the Washington environment.

Background

Introduction

Polychlorinated diphenyls (PCBs) are a class of persistent, bioaccumulative and toxic (PBT) compounds that were historically used in a wide range of consumer products. PCBs are created by reacting biphenyl with chlorine (Pomerantz, 1978). Historically, PCBs were used in electrical transformers and capacitors, heat transfer and hydraulic systems and vacuum pumps and lubricants, surface coatings, adhesives, plasticizers inks, insulating materials and pesticides (UNEP, 1999). Total worldwide production from 1929 to 1989 is estimated at 1.5 million tons of which 60% of worldwide and 77% of US production were used in the production of transformers of capacitors (Anderson, 2013). PCBs were valued for their persistence, inability to conduct electricity and anti-microbial effects. Companies have found alternatives for most PCB uses.

Total PCB loading to Puget Sound from the major pathways assessed by the Puget Sound Toxics Loading Analysis is as much as 2,000 kg/year. Releases from large capacitors is believed to account for 50% of the loading while a combination of large and small capacitors is thought to account for as much as 75% of the loading. Residential trash burning, transformers and sealants (caulking) are believed to account for the remaining loading at 13, 6 and 5%, respectively (Ecology, 2011c). Surface runoff accounts for the largest pathway to the Puget Sound, followed by wastewater treatment plants and air deposition. PCBs released to air and surface waters are delivered directly to Puget Sound. PCBs on land are mobilized during storm events and delivered to surface waters. PCBs are released in the highest quantities in commercial areas compared to other land covers, making PCB contamination especially relevant to the highly urbanized Puget Sound Basin (Ecology, 2011c).

Studies indicate that PCBs are ubiquitous throughout the natural environment, in air, soil and sediments, and are found in animals throughout the food chain (ATSDR, 2000). PCBs were detected in migrant Chinook salmon tissue and fish and marine mammals locally important to the Puget Sound region. Concentrations, however, appear to be declining in Puget Sound harbor seals and mussels. No equivalent trend can be identified in fish although modeling suggests levels will start to decline in English sole by 2020 (Ecology, 2011c). Due to PCB concentrations, fish consumption advisories have been issued for both marine and fresh water species in the Puget Sound basin. . In addition, salmon returning for spawning are also believed to be an additional albeit small source of PCBs to the Puget Sound region (Ecology, 2011c).

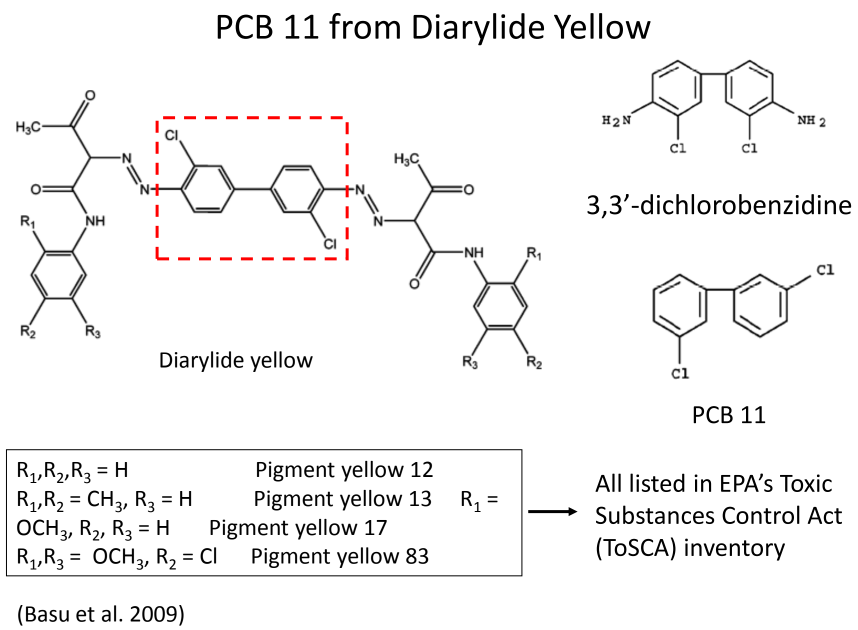
Historically, PCBs were manufactured in ninet major mixtures called Aroclors. Aroclor was the tradename of the technical mixture of PCBs that were sold in the United States by Monsato

Chemical Company. Prior to 1997, Monsanto Chemical Company in Sauget, Illinois produced approximately 99% of the PCBs used within the US. The nine Aroclor mixtures produced during this time period include Aroclors 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262 and 1268 (ATSDR, 2000). Production of these Aroclor mixtures decreased from over 86 million pounds in 1970 to 35 million pounds in 1977. (EPA, 2012a). Current releases are primarily due to a cycling of these historical amounts with slight additions from current uses and inadvertent sources. For example, EPA reports that from 1987 to 1993, PCBs released to land and water totaled slightly more than 74,000 pounds in the United States (EPA, 2012a), a fraction of the pre-ban use and releases. Inadvertent sources include contaminants or byproducts from manufacturing processes using chlorinated compounds either as a reaction component or solvent.

PCBs in products

Recent studies have shown that PCBs can still be found in products (Wu, 2010; Rodenburg, 2012). Many of these products contain PCBs as an impurity created during the production process. Building upon earlier EPA work, the New York Academy of Sciences identified 70 chemical processes that are likely to contain PCBs as contaminants from manufacturing processes (NYAS, 2005). Hu et al. conducted sampling of consumer paints containing specific azo (yellow) and phthalocyanine (blue and green) organic dyes and found PCB levels ranging from 2 to 200 ppb in 15 of 33 consumer paints tested. Diarylide yellow comprises approximately 25% of the 250 million tons of organic dyes produced yearly worldwide (Rodenberg, 2012) and testing has shown PCBs and especially PCB-11 are produced during its dye and pigment manufacture. As shown in Figure 1, PCB-11, indicated in the red box, is part of the structure of diarylide yellow. PCB-11 can either be produced as a byproduct during the manufacturing process or result from degradation of the dye.

Figure 1: Diarylide yellow and PCB-11 (From Rodenburg, 2012)



In addition to PCB-11, larger molecular weight PCBs (PCB-206, PCB-208 and PCB -209) are produced as a byproduct from the manufacture of the inorganic pigment titanium dioxide (TiO_4) (Rodenburg, 2012). Chlorine is reacted at high temperatures with titanium dioxide (TiO_2) containing ores to form titanium tetrachloride ($TiCl_4$) which is a liquid. $TiCl_4$ is then reacted with oxygen to make pure TiO_4 (UNEP, 2007). During this product process, the larger molecular weight PCBs are created as a reaction byproduct.

Ecology, in its assessment of chemical impacts to the Puget Sound, found PCBs in composite samples of caulk from buildings built between 1950 and 1978. Eight of 17 samples (47%) contained PCBs at concentrations of 0.77 to 920 mg/kg (Ecology, 2011c).
PCBs were added to joint sealant materials to improve their flexibility, increase their resistance to mechanical erosion, and improve adherence to other building materials (Diamond et al., 2010).

Project Description

Ecology's HWTR Program will conduct a study of consumer products both that are believed to contain PCBs as an active ingredient at the ppm level and those that may contain PCBs as a production impurity at the ppb level. The objectives of the study are to:

- Assess the levels of PCBs in general consumer products both for intentional and unintentional addition.
- Confirm the continued presence and potential non-point release of PCBs within Washington State.

Products will be purchased based upon information from previous studies showing presence of PCBs and from those products believed to contain PCBs as an active ingredient. Products found in existing studies (Wu, 2010; Rodenburg, 2012) to contain these PCBs include:

- PCBs as a contaminant (ppb level):
    - Paint.
    - Newspapers.
    - Yellow plastic bags.
    - Glossy magazines.
    - Cardboard.
- As an active ingredient (ppm level):
    - Caulk

Furthermore, additional sources will be searched to identify potential products containing unintended PCB contamination. 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, etc.) and sampling reports from authoritative bodies.

Emphasis will be placed upon products containing:

- Yellow, green or blue products using organic azo-dyes or pigments.
- White titanium dioxide.

Tthose products containing the highest levels of dyes or pigments will be prioritized. For example, product Material Safety Data Sheets (MSDS) that list high levels of TiO_2 will have priority over samples reported to contain lower levels. Samples possibly containing any of the PCBs of interest will be sent to a contract laboratory for analysis.

Sampling Process Design (Experimental Design)

Approximately 40 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 PCBs of interest as identified above.

Product Selection

Product selection will focus on products that historically have contained banned PCBs and products likely to contain the PCBs of interest based upon information gleaned from other resources and products and product components that are most likely to impact the Puget Sound.

Products will be selected that meet the following criteria:

1. Products that previously contained PCBs prior to the ban. An example is pre-1977 caulk that could contain as much as 100,000 ppm PCBs (EPA, 2012b).
2. Products that currently contain PCBs as an active ingredients such as caulk.
3. Products that can be shown to or are likely to contain either of the two dyes of interests.

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

- Peer-reviewed scientific articles that have tested products containing PCBs or other PCBs of interest found in consumer products. For example, Hu (2010), Rothenburg (2012) 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 PCBs in products that meet the above criteria.
- Caulks identified as currently containing PCBs as an active ingredient.

Products that will be considered for possible analysis include but are not limited to those shown in Table 1:

Table 1: Possible products for PCB analysis

Products	**Justification**
Cardboard containing yellow printing	Shown to contain PCBs as a contaminant[+]
Newspaper, particularly colored print such as the Sunday Comics	Shown to contain PCBs as a contaminant[+]
Paint	Shown to contain PCBs as a contaminant[++]
Caulk	Known to contain PCBs as a product component[+++]

[+] See Rothenburg, 2012. [++] See Hu, 2010. [+++] See EPA 2012b.

Target Chemicals

Target chemicals proposed for testing, and recommended practical quantitation limits (PQLs) for laboratory analysis of each, are shown in Table 2.

Table 2. Analytes of Interest

PCB	CAS	PQL^{+} (ppm^{++} or ppb^{+++})
3,3'-Dichlorobiphenyl ; PCB-11	2050-67-1	0.5^{+++}
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl; PCB-206	40186-72-9	0.5^{+++}
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl; PCB-208	52663-77-1	0.5^{+++}
Decachlorodiphenylether; PCB-209	2051-24-3	0.5^{+++}
PCBs[2]	1336-36-3	1.0^{++}

\+ The PQL are based upon the sediment PQLs reported in Method 1668 (EPA, 1999).
$^{++}$ ppm = parts per million of PCB congener by weight. $^{+++}$ ppb = parts per billion of PCB congener by weight.

PCBs are intentionally used in products at the part per million levels (ppm) and contaminant level, 1-200 part per billion, from products containing yellow organic and titanium dioxide pigments (Rodenberg, 2012; Hu, 2010) which are two to 400 times above the PQL. Samples containing the highest levels of PCBs of interest based on all available information such as product labels, product databases and other readily-available sources will be sent to the laboratory for analysis.

Organization and Schedule

Table 3 lists the individuals involved in the project and Table 4 contains a schedule.

Table 3. Organization of Project Staff and Responsibilities

Staff	Title	Responsibilities
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 and Client	Reviews project scope and budget, tracks progress, reviews draft QAPP and approves final QAPP.
Samuel Iwenofu (360) 407-6327	HWTR QA Coordinator	Reviews draft QAPP and recommends approval and reviews draft report.

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

[2] PCBs in the caulk samples are equivalent to the sum of the Aroclor mixtures and individual PCB congeners identified in EPA SW-846 method 8082A.

Table 4. Proposed Schedule for Completing Field and Laboratory Work and Reports

Field and laboratory work	Due date	Lead staff
Field work completed	April 2013	Alex Stone
Laboratory analyses completed	June 2013	
Final report		
Author lead / Support staff	Alex Stone	
Schedule		
Draft due to supervisor	July 2013	
Final (all reviews done)	August 2013	
Final report due on web	September 2013	

Sample Collection and Preparation

Products will be obtained in person or through internet retailers by HWTR staff. Samples will be prepared using the standard operating procedure developed for previous product sampling events (Ecology, 2012a). A brief summary of this procedure is described below.

All field and laboratory staff handling products 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.

Upon collection, products will be removed from their original packaging using pre-cleaned stainless steel implements. Products will be separated into to two 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 be a portions of the product (Fraction 2) that will be sent for PCB analysis.

For example, newspapers or magazines may be selected for analysis. Portions most likely to contain dyes or pigments of interest will be separated from the bulk product and sent for analysis. In the newspaper example, any color inserts including comics sections mostly likely to contain high concentrations of dyes will be separated from other portions of the paper and treated as an individual sample. 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. Cryomilling decisions will be made on a case-by-case basis. Non-plastic items such as paper, cardboard and textiles 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. Ecology will identify a company who can cryomill these samples.

Product samples will be sent to a contract laboratory for organic analysis. Photos and descriptive notes on each product purchased such as approximate thickness, surface roughness, material makeup, etc. will be recorded. Other information may be collected for the purpose of product and manufacturer identification.

Analytical Procedures

Laboratory

Appropriate methods will be used to measure extractable concentrations of all PCB congeners. . Appropriate methods will be suitable for the sample media that will minimize matrix interferences. These methods, along with estimated reporting limit (RLs), are listed in Table 5. At a minimum, sample extraction, cleanup and analysis shall follow the methods identified in EPA Method 1668C (EPA, 2010) or equivalent for the low level samples and EPA SW-846 Method 8082A or equivalent for the higher level caulk samples.

Table 5. Laboratory Methods and Reporting Limits

Analyte	Digestion Method	Instrumentation	Method	RL[+++]
Individual PCBs-low level	1668B	HR GC/MS[+]	EPA 1668C	1.0 (ppb)[++++]
Individual PCBs-caulk	3541	GC/MS[++]	EPA 8082	1 (ppm)[+++++]

[+]HR GC/MS = High Resolution Gas chromatography/mass spectroscopy [++] GC/MS = Gas chromatography/mass spectroscopy [+++]RL = Reporting Limit [++++]ppb = parts per billion of analyte in sample by weight (ppb)[+++++] = 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	40	$25.00	$1,000.00
PCBs-low levels	35	$700.00	$24,500.00
PCBs-higher levels	15	$300.00	$4,500.00
Total			**$30,000.00**

Quality Objectives

Quality objectives for this project are to obtain data of sufficient quality so that the amount of PCB contaminants can be determined from the following three sources:

1. Contaminants found in consumer products.
2. Intentionally added PCBs in consumer products at levels below the legal limit of 50 ppm.
3. PCBs found from caulk used in older buildings currently in use in Washington State.

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

MQOs for PCB analysis are shown in Tables 7. It is expected that the contract laboratory 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 Laboratory Analyses

Analyte	**Laboratory Control Samples** (recovery)	**Matrix**$^{+}$ **Spikes** (recovery)	**Duplicates**$^{+}$ (RPD)$^{++}$	**Method Blanks** (ppb)$^{+++}$	**Surrogate Recovery** (recovery)
Individual PCBs	85 - 115%	40-140%	± 25%	< 1.0	30-150%

$^{+}$ Matrix spike and split duplicates $^{++}$RPD = Relative Percent Difference $^{+++}$ppb = parts per billion

Quality Control Procedures

Field

No sampling will be conducted in the field. All samples will be purchased and brought back to Ecology for preparation prior to submittal for analysis; therefore, no field quality control procedures are anticipated for this project.

Laboratory

Table 8 displays the laboratory QC tests planned for PCB analysis. Laboratory QC tests will consist of laboratory control samples, matrix spikes, matrix spike duplicates, laboratory duplicates, and method blanks. Final PCB results will be corrected for surrogate recovery. The laboratory will test one methanol blank in addition to the method blank per batch of 20 samples processed.

Table 8. Quality Control Tests

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

LCS: Laboratory Control Sample Batch: maximum of 20 samples

Data Management Procedures

Data packages from the 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.

All data will be stored on the Reducing 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 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 bench sheets. 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 impact they may have upon the results of the study. This includes possible impacts on enforcement or any other use of the data.

Audits

The contracted laboratory will obtain accreditation by the State of Washington for analysis of the target PCBs by Method 1668. 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:

- Any deviations from the QAPP and sample preparation, QA/QC requirements, etc.
- Assessment of product test results from general consumer products for PCBs.
- Determination of what levels of specific PCBs are found in general consumer products.
- Data on specific products and product components found in Washington State.
- Appendices that include final SOP and tables showing results of laboratory analyses.

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Appendix A. Acronyms and Abbreviations

Following are acronyms and abbreviations used frequently in this report.

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 words
LCS	Laboratory control sample
LOQ	Limit of Quantitation
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
NEP	National Estuary Program
NICNAS	National Industrial Chemicals Notification and Assessment Scheme
OECD	Organisation for Economic Cooperation and Development
PCB	Polychlorinated biphenyls
PBT	persistent, bioaccumulative, and toxic substance
PPB	Parts per billion
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
W2R	Waste 2 Resources Program

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
ppb	parts per billion
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