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State of Washington

Quality Assurance Project Plan

Evaluation of Bisphenol A in Products Regulated by the State of Washington

June 2012

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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.

The plan for this study is available on Ecology's website at www.ecy.wa.gov/biblio/1203106.html.

Data for this project will be available upon request. Contact Joshua Grice, Waste 2 Resources Program, at 360-407-6786, with data requests.

Ecology's Activity Tracker Code for this study is 13-022.

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EAP: Environmental Assessment Program
SCS: Statewide Coordination Section

Table of Contents

List of Figures and Tables

Page

Figure

Tables

Abstract

In 2010, Washington State passed a law prohibiting the sale or distribution of certain products containing bisphenol A (BPA) (RCW 70.280). These products included: (1) bottles, cups, or other containers designed to be filled with liquid, food, or beverage and intended for children three years of age or younger and (2) sports bottles (effective July 2012). Metal cans less than 0.0149" thick are exempt from the ban.

This Quality Assurance Project Plan outlines a study to evaluate BPA in products affected by the law. Ecology's Environmental Assessment Program will collect and analyze approximately 85 product samples for BPA in July, 2012. A final report summarizing findings is anticipated in February, 2013.

Background

Bisphenol A (BPA) is a high production volume chemical used primarily in the manufacturing of polycarbonate plastics and epoxy resins. Many industries use polycarbonate to make products such as bottles, tableware, compact disks, and eyeglass lenses. Epoxy resins are used as protective linings on metallic food and beverage cans. BPA is also sometimes found in other types of resins, and in flame retardants, polyvinyl chloride plastic, dental fillings, and thermal papers.

BPA does not bioaccumulate, but it is widely present in humans due to its ubiquity in consumer products. In a representative study, Calafat et al. (2008) found detectable levels of BPA in 93% of the U.S. population over 6 years old. A primary pathway of human exposure is thought to be through BPA-containing food packaging (EPA, 2010). BPA can migrate into food and beverages from residual BPA in can linings and polycarbonate drinkware, or through hydrolysis of polycarbonate in drinkware (Aschberger et al., 2010). BPA migration from polycarbonate varies based on several factors, including contact time, temperature, and pH of the liquid (Aschberger et al., 2010).

Animal studies have shown BPA to be a reproductive, developmental, and systemic toxicant and weakly estrogenic (EPA, 2010). The National Toxicology Program (U.S. Department of Health and Human Services) stated "some concern" for effects in humans on the brain, behavior, and prostate gland in fetuses, infants, and children at current human exposures to BPA (NIH, 2008). Newborns and small children are thought to be at particular exposure risk, with studies estimating newborn BPA levels to be 11 times greater than adults (Edginton and Ritter, 2009).

Concern over the potential for adverse health effects from BPA exposure and the widespread occurrence of the chemical has prompted Canada, the European Union, and several U.S. states to take action against BPA in certain products.

In 2010, Washington State passed a law banning the sale of certain products containing BPA (RCW 70.280). The first ban, targeting products to be used by children under age three, became effective on July 1, 2011. These products include bottles, cups, and containers designed to be filled with liquid, food, or beverages. The second ban will take effect July 1, 2012, prohibiting the sale of any sports bottle up to 64 ounces that contains BPA. Metal cans less than 0.0149″ thick are not subject to the ban.

BPA is also included in the Child Safe Products Act (CSPA) Reporting List (WAC 173-334). Beginning in August 2012, manufacturers of children's products must report to Ecology if their products contain BPA.

Project Description

This project plan outlines a study to evaluate BPA levels in bottles, cups, and containers affected by the Washington State ban. The primary goal of the project is to assess compliance in products that are subject to the ban. The law does not specify a regulatory concentration level of BPA that is not to be exceeded. Instead the law states that the targeted products cannot be sold if the product "contains" BPA. In this project, BPA concentrations will be reported if exceeding 1 ppm (part per million).

Ecology's Environmental Assessment (EA) Program will collect and analyze approximately 85 product samples for BPA. Products will be collected in July 2012 and sent to a contract laboratory for analysis.

Product Selection

The EA Program will collect products of these types:

- baby bottles
- cups and containers, targeted to children less than 3 years old, used for liquid, food, or beverages
- sports bottles under 64 ounces

Figure 1 displays the anticipated breakdown of product types selected for analysis. A range of types of products will be collected. Approximately 75% of samples will be products targeted to children less than 3 years old (e.g., baby bottles and sippy cups) and 25% will be sports bottles. Products that appear to be made of polycarbonate or polycarbonate-like material will be targeted for collections.

Samples will be collected from a range of retailers but will focus on cheaper products, such as those sold in discount stores. Products labeled as "BPA-free" will be collected for verification purposes.

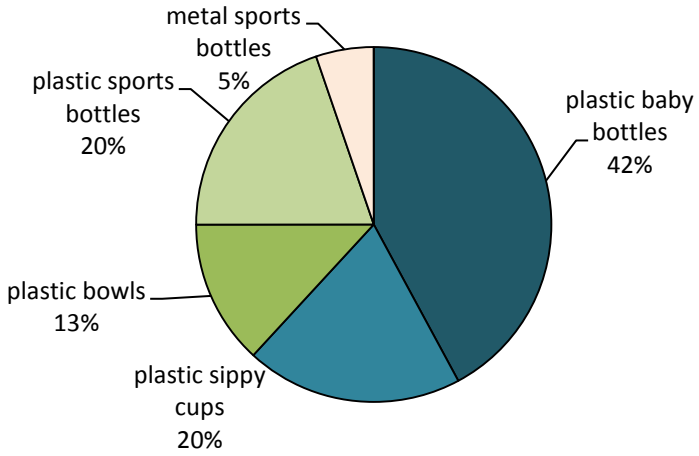

Figure 1. Anticipated Percentage of Product Types Collected for BPA Analysis.

Organization and Schedule

Table 1 lists the people involved in this project. All are employees of the Washington State Department of Ecology. Table 2 presents the proposed schedule for this project.

Table 1. Organization of Project Staff and Responsibilities.

Staff (all are EAP except client)	Title	Responsibilities
Joshua Grice W2RP Phone: 360-407-6786	Client	Clarifies scopes of the project. Provides internal review of the QAPP and approves the final QAPP.
Carol Kraege W2RP Phone: 360-407-6906	EAP Client	Clarifies scopes of the project. Approves the final QAPP.
Callie Mathieu Toxics Studies Unit SCS Phone: 360-407-6965	Project Manager and Principal Investigator	Writes the QAPP. Oversees product collection and transportation of samples to the laboratory. Conducts QA review of data, and analyzes and interprets data. Writes the draft report and final report.
Dale Norton Toxics Studies Unit SCS Phone: 360-407-6765	Unit Supervisor for the Project Manager	Provides internal review of the QAPP, approves the budget, and approves the final QAPP.
Will Kendra SCS Phone: 360-407-6698	Section Manager for the Project Manager	Reviews the project scope and budget, tracks progress, reviews the draft QAPP, and approves the final QAPP.
Dean Momohara Manchester Environmental Laboratory Phone: 360-871-8801	Acting Lab Director	Approves the final QAPP.
William R. Kammin Phone: 360-407-6964	Ecology Quality Assurance Officer	Reviews the draft QAPP and approves the final QAPP.

EAP: Environmental Assessment Program
QAPP: Quality Assurance Project Plan
SCS: Statewide Coordination Section
W2RP: Waste 2 Resources Program

Table 2. Proposed Schedule for Completing Field and Laboratory Work and Reports.

Field and laboratory work	Due date	Lead staff
Field work completed	July 2012	Callie Mathieu
Laboratory analyses completed	October 2012	
Final report		
Author lead	Callie Mathieu	
Schedule		
Draft due to supervisor	November 2012	
Draft due to client/peer reviewer	December 2012	
Final (all reviews done) due to publications coordinator	January 2013	
Final report due on web	February 2013	

Sample Collection and Preparation Procedures

EA Program staff will obtain products in person at stores near Olympia, WA, or through internet retailers selling to Washington State consumers. Upon collection, products will be brought back to Ecology headquarters and removed from their original packaging. Product handling will be done in Ecology's sample processing room on a clean bench lined with aluminum foil. All staff handling the product samples will wear powder-free nitrile gloves. Each product will be assigned a unique sample identification number and photos and descriptive notes will be recorded.

Product parts consisting of different material or function will be separated for individual analysis (e.g., bottle, ring, and nipple). To maximize the laboratory budget, not every component will be analyzed from each product. However, base or "bottle" parts consisting of polycarbonate or polycarbonate-like material will be sampled from each product. Other types of product parts are likely to vary in material and have a lower potential to contain BPA. These other product types will be sampled at least once during the project.

Staff will reduce the product part in size by cutting into smaller pieces with stainless steel tools. All tools used for size reduction will be decontaminated between samples using standard procedures. The following decontamination procedure will be followed: hot water scrub with Liquinox soap, deionized water rinse, acetone rinse, and hexane rinse. Acetone is used to remove any trace soap residue and hexane is used to remove any trace organic compounds.

Samples reduced in size will be wrapped in aluminum foil and placed in 8 oz. glass jars prior to being sent to the contract laboratory for analysis. Samples will be shipped to the laboratory at ambient air temperature, to reduce the potential for contamination through wet or blue ice. Chain of custody procedures will be followed throughout the process.

Analytical Procedures

The contract laboratory will measure BPA using LC/MS/MS according to EPA Method 1694. Products will be cryomilled prior to analysis, as the method states that particles must be in the form of finely divided solids for efficient extraction. Cryomilling also provides a homogenous sample from which an aliquot will be taken. Sample aliquots will undergo microwave-assisted solvent extraction. After extraction, samples are concentrated, reconstituted in water, and cleaned up using solid phase extraction cartridges.

This analysis is aimed at achieving a "total" concentration of BPA in the product, as opposed to a leaching or migration test. Manufacturers are prohibited from selling products that *contain* BPA, which is analogous to a total concentration. Table 3 describes the digestion and analysis methods along with estimated reporting limits.

Table 3. Laboratory Methods and Reporting Limits.

Analyte	Extraction	Analysis	Method	Reporting Limits (ppm)
Bisphenol A	Microwave-assisted solvent extraction	LC/MS/MS	EPA 1694	1.0

LC: liquid chromatography
MS: mass spectrometry
ppm: parts per million

Budget

The product collection and laboratory costs estimated for this project totals $46,200. Table 4 displays the laboratory costs associated with this project.

Table 4. Laboratory Budget.

Item	Number of Samples	Cost per Sample	Total Cost ($)
Product collection	40	15	600
Laboratory prep. and analysis*	85	456	38,760
Laboratory QC samples	15	456	6,840
		Total project costs:	46,200

*Includes $90 charge per sample for cryomilling and 25% MEL contract fee.

Quality Objectives

The quality objective for this project is to obtain data of sufficient quality for confident quantification of BPA levels in plastic and metal matrices. Uncertainties will be minimized through careful attention to the sampling, measurement, and quality control procedures outlined in this project plan.

Measurement Quality Objectives

Table 5 presents the measurement quality objectives (MQOs) for laboratory analysis of BPA. The contract laboratory is expected to meet these criteria. The project manager will review data falling outside of the acceptance limits for their usability. Data will be appropriately qualified when necessary.

Table 5. Measurement Quality Objectives.

Analyte	Laboratory Control Samples (recovery)	Matrix spikes (recovery)	Duplicates (RPD)	Method Blanks (ppm)	Surrogate (recovery)
Bisphenol A	60 - 140%	40 - 140%	≤ 30%	< 1	25 - 150%

RPD: relative percent difference
ppm: parts per million

Quality Control Procedures

Field

No field quality control procedures (QC) are planned for this project.

Laboratory

Table 6 displays the laboratory QC tests planned for BPA analysis. Laboratory QC tests will consist of laboratory control samples, matrix spikes, matrix spike duplicates, laboratory duplicates, and method blanks. TestAmerica will use BPA-d6 as an internal surrogate for every sample, per isotopic dilution technique. Final BPA results will be corrected for surrogate recovery.

The cryomill vessel used in sample preparation will be rinsed with deionized water and methanol between each sample. The laboratory will test one methanol blank per batch of 20 samples processed. This is separate from the method blank, which is taken through the entire process, including cryomilling preparation.

Table 6. Laboratory Quality Control Tests.

LCS	Matrix Spikes	Matrix spike Duplicates	Laboratory Duplicates	Method Blanks	Surrogate Recovery
1/batch	1/batch	1/batch	1/batch	1/batch	every sample

LCS: Laboratory Control Sample
batch: maximum of 20 samples

Data Verification

MEL will review the data packages from the contract laboratory. Data packages will include Tier III deliverables such as calibration reports, chromatograms, and spectra benchsheets. MEL 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.

MEL 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 Management Procedures

Product notes, purchase receipt copies, and photos of products will be stored by the project manager. No publicly-available database currently exists for analytical data on products tested by Ecology. This type of data storage is anticipated for the future. In the interim, data for this project will be available upon request to Joshua Grice, at 360-407-6786.

Audits and Reports

Audits

The contract laboratory will obtain accreditation by the State of Washington for BPA analysis by Method 1694. 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.

Reports

A final report for this project will be published after an internal review period. The final report will include:

- Categorical descriptions of the products analyzed. Product names, brands, and retailers will not be named in the final report.
- Laboratory results of BPA analysis (with no product name associated to the result).
- Statistical summary of BPA concentrations measured for this project.
- An assessment of compliance with the state ban.

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Appendix. Glossary, Acronyms, and Abbreviations

Glossary

Bioaccumulative: Accumulated by animals and humans and increase in concentration up the food chain.

Cryomilling: The process of reducing a sample to very small particle sizes by employing cryogenic temperatures and a mechanical mill.

Hydrolysis: A chemical reaction in which a compound reacts with water to produce other compounds.

Acronyms and Abbreviations

BPA	Bisphenol A
CSPA	Children's Safe Products Act
EA	Environmental Assessment
Ecology	Washington State Department of Ecology
EPA	U.S. Environmental Protection Agency
et al.	And others
LCS	Laboratory Control Samples
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
QA	Quality assurance
QC	Quality Control

Units of Measurement

°C	degrees centigrade
cm	centimeter
ppm	parts per million