

Quality Assurance Project Plan

Metals in Packaging

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Publication Information

It is Washington State Department of Ecology policy to have an approved Quality Assurance Project Plan for all Agency-sponsored sampling events. 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 a report of the study to the Internet.

The plan for this study is available on the Department of Ecology's website at https://apps.ecology.wa.gov/publications/SummaryPages/1504014.html.

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May 2015

Approved	by:
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HWTR - HQ: Hazardous Waste and Toxics Reduction Program - Headquarters

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Abstract

The Washington State Department of Ecology's (Ecology) Product Testing Program is conducting an ongoing study to determine compliance with Washington's Toxics in Packaging Legislation passed in 1991. This legislation places restrictions on four metals in packaging, including mercury, cadmium, lead, and hexavalent chromium. As of 1995, products may not be sold in Washington State that contain these four metals either individually or in total above 100ppm. Eighteen additional states have passed similar toxics in packaging legislation.

In 2007, Washington State became a member of the Toxics in Packaging Clearinghouse (TPCH), an association of nine states with similar toxics in packaging legislation. The TPCH coordinates implementation of state legislation on behalf of its member states, with the goal of promoting consistency across states. It is a resource and single point of contact for companies seeking information on or an exemption from toxics in packaging requirements.

The TPCH also assists member states in enforcing toxics in packaging requirements and coordinates product testing across member states. Since becoming a TPCH member, Washington has participated in several product testing initiatives, including testing PVC packaging, plastic bags, shopping bags, metal packaging components and glass to mention a few. TPCH's sampling and enforcement efforts have been very successful in educating industry on toxics in packaging requirements, increasing compliance with packaging requirements, and significantly reducing the amount of toxic metals used in consumer product packaging.

In this study, Ecology will screen approximately 300 samples per year of packaging purchased from other product testing studies, packaging donated from staff, and packaging from products purchased specifically for the packaging. From the screened samples, Ecology will select up to 50 samples per year for laboratory analysis of total mercury, cadmium, lead, and chromium.

The results from the testing will be posted to the **Ecology Product Testing Database**.

Background

Metals

In 1991, the Washington State Legislature passed Chapter 70.95G RCW (Packages Containing Metals, 1991). This legislation limits the amount of four toxic metals (mercury, cadmium, lead, and hexavalent chromium) in packaging sold in Washington State. Ecology was identified as the responsible agency for implementing this legislation. The legislation contains a very broad definition for both packaging and packaging components¹. Packaging is defined as:

"Package" means a container providing a means of marketing, protecting, or handling a product and shall include a unit package, an intermediate package, and a shipping container. "Package" also means and includes unsealed receptacles such as carrying cases, crates, cups, pails, rigid foil and other trays, wrappers and wrapping films, bags, and tubs.

A packaging component is defined as:

"Packaging component" means an individual assembled part of a package such as, but not limited to, any interior or exterior blocking, bracing, cushioning, weatherproofing, exterior strapping, coatings, closures, inks, and labels.

The legislation establishes a limit of 100 ppm for the <u>total</u> concentration of all four metals or for any metal individually. Ecology does not have penalty authority under the legislation but may ban the sale of any product that does not meet the regulated levels if a company refuses to comply.

In 2007, Ecology joined the Toxics in Packaging Clearinghouse (TPCH), an association of nine states with similar legislation². The TPCH has facilitated education and outreach to businesses on toxics in packaging requirements and has conducted several sampling events to emphasize the need for compliance with packaging legislation. Individual states have also conducted packaging sampling to guarantee compliance.

Project Description

The objective of the study will be to determine compliance with the Washington State toxics in packaging legislation.

Ecology's Product Testing Program will conduct a study that screens for cadmium, mercury, chromium, and lead in packaging with a portable XRF analyzer. Samples that contain sufficient metals of interest will be sent to Manchester Environmental Laboratory or a contract lab for analysis. Samples will be prepared and screened using procedures identified in the Product Sampling Standard Operating Procedure (Ecology, 2015).³

¹ 70.95G.010, accessed 11/05/2014.

² Toxics in Packaging Clearinghouse website available at: http://www.toxicsinpackaging.org/, accessed 2/10/2015.

³ The final Standard Operating Procedures is currently under review. If you have questions, please contact <u>Saskia van Bergen</u> at 360-407-6609.

Sampling Process Design (Experimental Design)

About 300 packaging samples from consumer products will be screened annually. Product packaging from other product sampling Quality Assurance Project Plans (QAPPs) may be considered for analysis. Additional packaging samples may be purchased or obtained from local stores and internet retailers for testing. Emphasis will be placed on specific types of packaging that have been found to be problematic in the past.

All packaging samples will be screened with a portable XRF for the metals of concern to determine if laboratory analysis is necessary. Each year, approximately 25-50 of these screened packaging samples will be forwarded for metals analysis if they appear to violate compliance screening criteria. Metal analysis will be completed by inductively coupled plasma mass spectroscopy (ICP-MS).

Packaging Selection

Consumer products selected for analysis will mainly focus on specific types of packaging found by the TPCH to be an issue in previous studies, including but not restricted to: soft vinyl plastic, packaging for specific uses, such as reusable bags, and certain dyes and inks. Glass and metal components may also be screened. Screening of other packaging materials will be performed on a less frequent basis.

Packaging Screening

Packaging will be screened using a portable XRF analysis to assist in the identification of samples that are likely above the level of compliance.

Target Chemicals

Chapter 70.95G RCW establishes a limit of 100 ppm for the <u>total</u> concentration of mercury, lead, cadmium, and hexavalent chromium.

For screening purposes, packaging containing 75 ppm individually of mercury, lead, or cadmium or packaging containing a total of mercury, lead, and cadmium greater than 100 ppm will be forwarded to the laboratory for validation (to the limits of the project budget). A few samples with elevated levels of total chromium will also be selected. In the instance where there are more detectable levels of metal than the budget will allow, those packaging samples with the highest concentrations will be prioritized for analysis.

Samples sent to the laboratory will be analyzed for all four metals. The exact number of samples will depend on the availability of applicable packaging and budgetary constraints.

Organization and Schedule

Table 1 lists the individuals involved in the project and Table 2 contains a schedule.

Table 1. Organization of Project Staff and Responsibilities

Staff	Title	Responsibilities
Joel Bird	Manchester	Reviews draft QAPP.
Manchester	Environmental Lab	
Environmental Lab	Director	
Phone: 360-871-8801		
Joshua Grice, W2R	Product Testing	Reviews project scope, budget and tracks
(360) 407-6786	Coordinator	progress.
Samuel Iwenofu	HWTR QA	Reviews draft QAPP and approves final QAPP.
HWTR-HQ	Officer	
(360) 407-6964		
Saskia van Bergen	Project	Writes QAPP. Coordinates with laboratory.
HWTR-HQ Program	Manager/Client	Oversees product collection, processing and
(360) 407-6609		transportation of samples to laboratory.
		Conducts QA review of data, analyzes and
		interprets data.
Christina Wiseman,	Sampling Lead	Purchases products, conducts XRF screening
HWTR-HQ Program		of products and sends samples to laboratory.
360-407-7672		Enters data into Product Testing Database.
Ken Zarker, HWTR-HQ	Section Manager	Reviews project scope and budget, tracks
(360) 407-6698	for the Project	progress, reviews draft QAPP and approves
	Manager	final QAPP.

HWTR-HQ: Hazardous Waste and Toxics Reduction Program-Headquarters.

QAPP: Quality Assurance Project Plan.

W2R: Waste 2 Resources.

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

Lead staff: Saskia van Bergen

Sample collection and laboratory work	Due date
Sample collection	Ongoing
Laboratory analyses	2 months after received

Lead staff: Saskia van Bergen, support staff: Christina Wiseman

Reporting to database	Due date
Review/Qualify data	1 month after receipt of data
Upload to database	1 month after reviewed
Data publically available	Annually
Report (optional)	Annually

Sample Collection and Preparation

Products will be obtained in person or through internet retailers. In addition, packaging reserved from other Ecology sampling events will be evaluated to determine if they meet the requirements of this QAPP.

The packaging will be sampled according to the *Product Sampling Standard Operating Procedure* (Ecology 2015).⁴ Briefly, the samples will be screened for metals using a portable XRF and results entered into the Product Testing Database. Photos of each package screened will be recorded. Packages that contain appreciable levels of metals will be cut into approximately 1.5 cm² pieces and sent to Manchester Laboratory for analysis. Depending on the material, some samples might need to be milled. Laboratories under contract to the state to provide analytical data will be the back-up for analytical support. The Project Manager will be responsible for the review and evaluation of all laboratory analyses.

Analytical Procedures

XRF Analysis

Individual components of packaging will be screened using a Niton XL3t portable XRF analyzer (Figure 1) following the instrument manufacturer recommendations and procedures described in Ecology's Product Testing Procedures (Ecology 2015).⁵



Figure 1. Niton Portable XRF

⁴ The final Standard Operating Procedures is currently under review. If you have questions, please contact <u>Saskia van Bergen</u> at 360-407-6609.

⁵ The final Standard Operating Procedures is currently under review. If you have questions, please contact <u>Saskia van Bergen</u> at 360-407-6609.

Laboratory

Table 6 describes digestion and analysis methods along with estimated LOQ's. Metals samples will be prepared following EPA 3052 (microwave complete digestion) and measured using the Environmental Protection Agency's 6020A (ICP-MS). HF needs only to be used for glass matrices.

Table 3. Laboratory Methods and Reporting Limits

Analyte	Digestion Method	Instrumentation	Method	RL (ppm)
Cadmium	EPA 3052*	ICP-MS	6020A	1.0
Chromium	EPA 3052*	ICP-MS	6020A	1.0
Lead	EPA 3052*	ICP-MS	6020A	1.0
Mercury	EPA 3052*	ICP-MS	6020A	1.0

ICP-MS = Inductively-coupled plasma/mass spectrometry

RL = Reporting Limit

ppm = parts per million

Budget

The project budget is included in Table 7.

Table 4. Project Budget

	# of Samples	Cost per sample	Total
New Product Packaging	60	\$20.00	\$1,200
Metals	25-50	\$100.00	\$2,500-5,000
Metals (using HF)	0-20	\$145.00	\$0-\$2,900
Total			\$3,700-\$7,200

Quality Objectives

Quality objective for this project is to obtain data of sufficient quality so that the amount of metals in packaging from consumer and children's products can be determined. This will be achieved through careful attention to the sampling, sample processing, measurement, and quality control (QC) procedures described in this plan.

Measurement Quality Objectives

At a minimum, an XRF reading will be taken at the beginning and end of each 8-9 hour period on standards provided by the manufacturer. The standard chosen should be the material most similar to the sample matrix being analyzed. Since the XRF analysis is being used as a screening

^{*}HF is used only for glass matrices. Refer to the <u>TPCH Guidance on Laboratory Analysis for Toxics in Packaging</u> and <u>TPCH Guidance on analysis of Glass Matrices for Toxics in Packaging</u> for more information. Note: Glass samples will be contracted out.

tool only, no measurement quality objectives (MQOs) are outlined but the performance criteria in the SOP (Ecology 2015)⁶ should be met.

MQOs for laboratory analysis of metals are shown in Table 8. 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 5. MQOs for Laboratory Analyses

	Laboratory Control Samples	Matrix Spikes	Duplicates*	Method Blanks*
	(recovery)	(recovery)	(RPD)	(ppm)
Cadmium	85- 115%	75-125%	$\pm 20\%$	1.0
Chromium	85- 115%	75-125%	$\pm 20\%$	1.0
Lead	85- 115%	75-125%	$\pm 20\%$	1.0
Mercury	85- 115%	75-125%	$\pm 20\%$	1.0

^{*} Metals reporting limits were established by raising soil limits by a factor of 10

RPD – Relative Percent Difference

ppm = parts per million

Quality Control Procedures

Field

No field quality control procedures are anticipated for this project.

Laboratory

Table 6 shows laboratory QC samples planned per batch of 20 samples processed.

Table 6. Quality Control Tests

	Laboratory	Matrix	Matrix Spike	Laboratory	Method
	Control Samples	Spikes	Duplicates	Duplicates	Blanks
Elements	1/batch	1/batch	1/batch	1/batch	1/batch

^{*}Dependent on amount of sample available.

Spikes are at 100 ppm.

⁺Matrix spike duplicates and split duplicates

⁶ The final Standard Operating Procedures is currently under review. If you have questions, please contact <u>Saskia van Bergen</u> at 360-407-6609.

Data Management Procedures

XRF data and the NDT (Niton Data Transfer) files from the screening portion of the project will be uploaded to the Ecology Product Testing Database.

Data packages from MEL or a 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 should address condition of the samples on receipt, sample preparation, methods of analysis, acids used, instrument calibration, if the sample was completely digested, 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. The case narratives will be uploaded into the Ecology Product Testing Database.

Audits

MEL and any contract lab must participate in performance and system audits of their routine procedures. Results of these audits must be made available on request.

Report

If the products are part of a special study, a final report detailing the findings of the study will be completed through the Toxics in Packaging Clearinghouse. The final report will include:

- Categorical descriptions of the packaging screened with the portable XRF (brands, product names, etc. will not be included).
- Comparison of laboratory results with XRF screenings, where applicable.
- Summarized results based on product material.

Data Verification

The Project Manager will review all laboratory data generated by MEL and contract laboratories. The Project Manager will verify methods and protocols specified in this QAPP were followed: all calibrations, checks on quality control, and intermediate calculations were performed for all samples; and the data is consistent, correct, and complete, with no errors or omissions. Evaluation criteria will include the acceptability of procedural blanks, calibration, matrix spike recoveries, duplicates, laboratory control samples, and appropriateness of data qualifiers assigned.

A case narrative will meet the requirements for a data verification report for MEL's chemical data.

Data Quality (Usability) Assessment

The Project Manager will examine the data reviews, case narratives, and data packages to assess the usability of the data. To determine if project MQOs have been met, results for laboratory control samples, sample duplicates, matrix spikes, and internal standard recoveries will be

compared to QC limits. The method blank results will be examined to verify there was no significant contamination of the samples. To evaluate whether the targets for reporting limits have been met, the results will be examined for "non-detects" and to determine if any values exceed the lowest concentration of interest. Based on these assessments, the data will be either accepted, accepted with appropriate qualifications, or rejected and re-analysis required.

References

EPA Method 3052 (1996) Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3052.pdf

EPA Method 6020A (2007).

http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/6020a.pdf

Packaging Containing Metals, 1991. Chapter 70.95G RCW, available at: http://apps.leg.wa.gov/RCW/default.aspx?cite=70.95G&full=true, accessed 10/2/2014.

Glass Matrix Test Methods Evaluation for Toxics in Packaging, 2014 Toxics in Packaging Clearinghouse, accessed 4/8/15.

<u>Laboratory Round Robin Test Project: Assessing Performance in Measuring Toxics in Packaging</u>, 2011, Toxics in Packaging Clearinghouse, accessed 4/8/15.

Appendix A

Glossary, Acronyms, and Abbreviations

Following are acronyms and abbreviations used frequently in this report.

- **Ecology**: Washington State Department of Ecology
- **EPA:** Environmental Protection Agency
- **HQ**: Headquarters
- **HWTR:** Hazardous Waste and Toxics Reduction Program
- **MEL**: Manchester Environmental Laboratory
- MQO: Measurement quality objective
- ppm: parts per million
- QA: Quality assurance
- QAPP: Quality Assurance Performance Plan
- **RCW**: Revised Code of Washington
- **RPD**: Relative percent difference
- **SOP:** Standard operating procedures
- **W2R:** Waste 2 Resources Program
- WAC: Washington Administrative Code
- XRF: X-Ray Fluorescence