DEPARTMENT OF ECOLOGY
State of Washington

Quality Assurance Project Plan

Cadmium and Metals in Children's Jewelry

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Quality Assurance Project Plan

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October 2015

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CSPA: Children's Safe Product Act
HWTR: Hazardous Waste and Toxics Reduction Program
EAP: Environmental Assessment Program

1.0 Table of Contents

2.0 Abstract

The Washington State Department of Ecology (Ecology) will conduct a study in 2015 to evaluate children's jewelry for cadmium, lead, and the five additional metals (antimony, arsenic, cobalt, mercury, and molybdenum) listed as Chemicals of High Concern to Children (CHCC) under the Children's Safe Products Act (CSPA).

Toxic metals may be present in children's products as a result of engineering processes of metal alloys, or their use as an additive or an ingredient in a pigment or from using recycled metals stocks. The presence of toxic metals in children's products is a concern because several studies have reported evidence of carcinogenicity, as well as demonstrated reproductive, developmental, or neurological effects.

The CSPA has established limits on the levels of cadmium, lead, and phthalates allowed in children's products sold in Washington. The law also requires manufacturers to report to Ecology any children's product that contains CHCC, as established by Ecology and the Washington State Department of Health (DOH). Ecology regularly performs studies to assess manufacturer and product compliance with Washington laws.

During the fall of 2015, Ecology will procure approximately 250 items of children's jewelry designed for children 12 years old and younger. The items of jewelry will be disassembled into components and then screened for metals with an X-ray fluorescence analyzer (XRF) to aid in sample selection. Individual jewelry components will be submitted for laboratory analysis for the total element content of each of the seven target metals: cadmium, lead, arsenic, antimony, cobalt, mercury, and molybdenum.

3.0 Background

The Children's Safe Products Act (CSPA) was passed into law by the Washington State legislature in 2008. The law restricts cadmium and lead levels to 40 ppm and 90 ppm, respectively, in children's products. The law additionally mandated that the Departments of Ecology (Ecology) and Health (DOH) develop a list of chemicals of high concern to children (CHCC). Beginning in August 2012, manufacturers were required to report to Ecology if a children's product contains a chemical from this list. For more information on the CSPA legislation, visit http://www.ecy.wa.gov/programs/swfa/cspa/.

Ecology regularly conducts studies to ensure manufacturer compliance with the CSPA legislation. An assessment of children's jewelry for cadmium, lead, and CHCC metals has been prioritized due to increased concerns and the risk to children. A statement by the Chairman of the US Consumer Product Safety Commission (CPSC) illustrates a poignant recommendation and provides background for such worries about potential exposure to toxic metals in jewelry:

> *"Do not allow young children to be given or to play with cheap metal jewelry, especially when they are unsupervised. We have proof that lead in children's jewelry is dangerous and was pervasive in the marketplace. To prevent young children from possibly being exposed to lead, cadmium or any other hazardous heavy metal, take the jewelry away."* (Tenenbaum, 2010)

3.1 Study approach

This study will focus on the following jewelry designed for and to be worn by a child 12 years and younger: anklets, arm cuffs, bracelets, brooches, chains, crowns, cuff links, decorated hair accessories, earrings, necklaces, pins, rings, and body piercing jewelry[1], or any bead, chain, link, pendant, or other component of such an ornament.

The aforementioned jewelry items were further categorized into seven jewelry groupings for the procurement plan. Table 1 illustrates the jewelry groupings and targeted purchasing design.

The following criteria were used for delineating jewelry item type into groupings and defining the distribution for purchasing.

- Due to design or physical placement during adornment, more likely to come in contact with a child's mouth (accidently or purposefully).
- Widely abundant to consumers (e.g., higher proportion available on retail shelves).
- Available in large enough quantity for analysis (i.e., size of individual item of jewelry, or multiple pieces of an item of jewelry).

[1] Body piercing jewelry includes items specifically designed for new ear piercings, in addition to piercings in other areas of the body. It is not expected that the study will encounter "other" body piercing jewelry marketed to children; if encountered, it will be included in the study.

Table 1. Targeted Purchasing Plan.

Jewelry Product Groupings							Total Number of Samples
Chain or Necklace	Earrings	Armcuff or Bracelet	Rings	Brooches, cufflinks, pins, other ornaments	Body Piercing Jewelry	Hair Accessory, Crown, or Tiara	
45	45	45	45	30	30	10	250

3.1.1 Logistical problems

There may be difficulty in obtaining a large enough sample for laboratory testing of an item or an individual component of an item of jewelry. Products will be purchased in multiples and similar jewelry components combined in order to obtain samples large enough for testing.

3.1.2 History

Jewelry is designed using a variety of materials and is universally fashioned as an object of adornment. Higher quality jewelry may contain one or more types of precious metal and may be accompanied by precious or semiprecious gemstones. “Cheap” or inexpensive jewelry might be fashioned entirely of metal or even plastic, or contain components of these. The metals and the quality of metals can vary widely, and they can be adorned with inexpensive gems and stones, plastic beading, or be painted to add decorative attributes.

A silvery-white ductile metal, cadmium is rarely found in its pure form, but rather most commonly found in zinc ores, and to a lesser degree in copper and lead ores. The availability of cadmium can often be tied to the abundance of zinc, since cadmium is produced as a byproduct in the recovery of zinc metal from ores. As a dense but soft element, cadmium can be intentionally integrated into a metal alloy to enhance strength, and the soft nature allows for casting of intricate and decorative shapes. Electroplating with cadmium has served as effective for inhibiting corrosion and for base coating for paint applications. Additionally, coatings of cadmium on exterior surfaces can produce a polished appearance, providing an added decorative appeal and giving cheap jewelry a more expensive look, a desired quality in jewelry-making.

For centuries lead was widely used in fashioning decorative objects, castings, pipes, and as pigments (lead oxides) or in glazes on pottery. Much of the appeal of lead is due to the dense metal’s availability, low melting point, high malleability, and corrosion-resistant qualities. Mostly abundant as alloys, lead is commonly used in combination with zinc, copper, tin, and in some instances with antimony and arsenic.

Cobalt is used in fashioning objects for its desirable qualities such as corrosion resistance, tremendous strength, and a shiny bright white appearance, much like that of platinum. Increasingly cobalt is becoming a cost-effective and durable alternative material to titanium or platinum for manufacturing jewelry.

Cadmium and lead can serve as chemical stabilizers or softening additives in plastics. Pigments have long been derived from metals. Antimony, arsenic, lead, mercury, cadmium, cobalt, and molybdenum can produce colors that span the spectrum from white, hues of yellows to reds, brilliant greens, to vivid blues and violets. These pigments have been widely used for paints and incorporated into plastic products. Cobalt, molybdenum, and mercury compounds are used as catalysts in processing materials in the plastics industries.

3.1.3 Contaminants of concern

This study will evaluate the metals of high concern to children in jewelry manufactured for children. Headquarters Ecology personnel will use an XRF to screen individual components of jewelry for cadmium, lead, mercury, arsenic, antimony, cobalt, and molybdenum to assist in selection of samples for laboratory analysis.

3.1.4 Results of previous studies

Ecology began independently testing children's and consumer products in 2012 to assess manufacturer and retailer compliance with CSPA legislation and other consumer product laws. Metals were tested in children's products and packaging and in the tiered product compliance assessment projects by Stone (2014a,b) and Mathieu and Bookter (2014). Ongoing studies by Mathieu and McCall (2014) and Stone (2015a,b) investigating children's accessories and seasonal-based products will provide guidance on current market product availability.

Evaluation of the effectiveness of using an XRF as a screening tool is an ongoing practice in all consumer product testing studies. XRF data from this project will be added to the data pool for future evaluations.

3.1.5 Regulatory criteria or standards

The Children's Safe Products Act requires manufacturers of children's products to report on the presence of CHCC in their products. (Chapter 173-334 WAC). CHCC include toxic chemicals that have been documented to be present in the hair, blood, and urine of Washington residents, or they have been found in children's products. Currently, sixty-six toxic chemicals, including seven metals, have been collectively defined by Washington State Departments of Health and Ecology for inclusion to the CHCC list (Ecology, 2011a).

The reporting rule requires manufacturers of children's products to notify Ecology if a product component contains CHCC in any concentration greater than the practical quantitation limit (PQL) (Ecology, 2011b and Ecology, 2013). CHCC metals and mercury are reported when concentrations are 1.0 ppm and 0.5 ppm or higher, respectively, when the chemical was intentionally added to the product (Ecology, 2012). Notification is also required when a product component contains a metal of concern at a concentration of 100 ppm or higher and the manufacturer has identified the chemical as a contaminant.

Data reported by manufacturers on CHCC in their products is available to the public (https://fortress.wa.gov/ecy/cspareporting/).

As of August 2015, over 27,000 product components have been reported to contain a CHCC. To date, only 3.7 percent (1009) of products components entered are designated as jewelry components, and only 380 jewelry components have been reported to contain a CHCC metal[2], as illustrated in Figure 1. With the exception of cobalt, each metal was most frequently reported by manufacturers under the category of no function or contaminant. Cobalt was listed most often as a coloration/pigment/dye/ink.

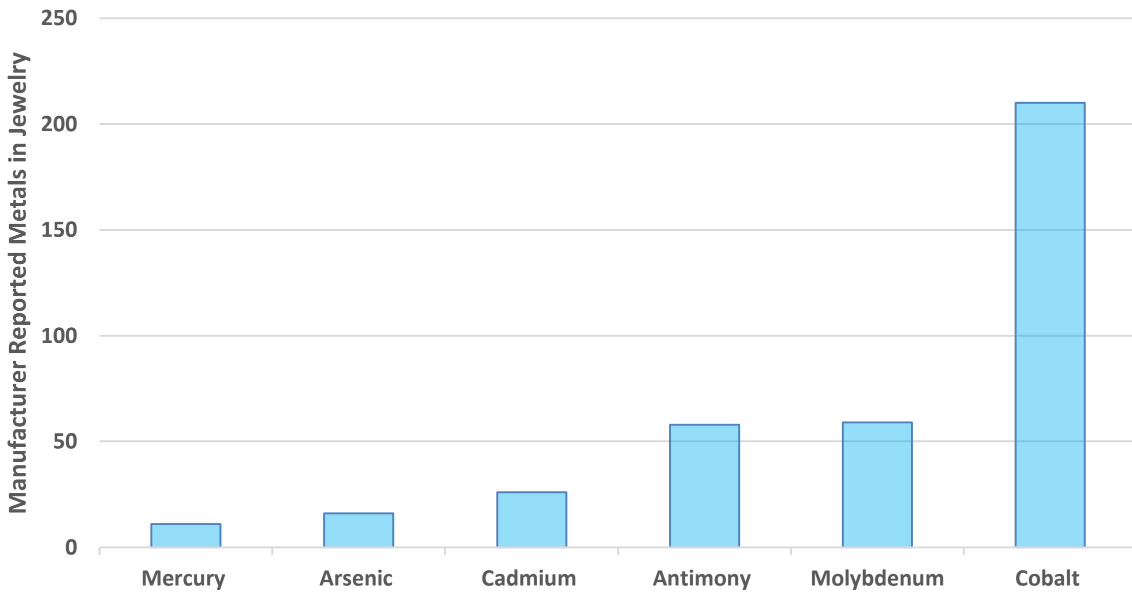

Figure 1. Manufacturer-Reported Metals in Jewelry Components.

[2] Lead was not included on the list of CHCC for manufacturing reporting due to preemption by federal law.

4.0 Project Description

Ecology will conduct a study to test for the presence of cadmium and CHCC metals[3] in children's jewelry. During the fall of 2015, approximately 250 samples will be purchased, deconstructed into components, and screened by an XRF. The XRF results will be reviewed to aid in the selection of samples for laboratory analysis. Those samples then will be prepared and sent to Manchester Environmental Laboratory (MEL) for analysis.

4.1 Project goals

This study is being carried out to:

- Assess the presence of total cadmium and CHCC metals in children's jewelry through quantitative laboratory analysis.
- Provide data to Ecology's CSPA Enforcement Officer to determine compliance with CSPA and the Manufacturer's Reporting Rule.

4.2 Project objectives

To meet project goals, Ecology staff will carry out the following objectives:

- Purchase and conduct XRF screenings on approximately 250 articles of children's jewelry bought from on-line and in-person retailers.
- Select 140 product components, with the aid of XRF measurements, for laboratory analysis of metals.

4.3 Information needed and sources

The CSPA manufacturer reporting database will be reviewed prior to product collection. A literature review of existing product testing data and previous studies on children's jewelry will also be completed to help provide a basis for product collection.

4.4 Target population

Products targeted in the study will be limited to children's jewelry. Items of jewelry are defined as those which are principally designed and intended to be worn as an ornament. Children's jewelry is further defined to be worn by children 12 years old and younger.

[3] The reference to "CHCC metals" will hereinafter include lead.

4.5 Study boundaries

Ecology staff will purchase products “off the shelf” from Puget Sound area stores and through on-line retailers. Large chain retailers and discount stores will be mainly targeted. The practice of statewide distribution by most of the retail chain stores ensures that products purchased from Puget Sound area stores are representative of products sold across the state.

4.6 Tasks required

Tasks to be performed for this study:

- Research children’s jewelry products on the Internet.
- Research prevailing laws, testing methods, and previous studies.
- Purchase children’s jewelry.
- Enter products and product components into the Product Testing Database (PTDB).
- Deconstruct jewelry into components for testing.
- Screen product components by the XRF for the presence of cadmium and CHCC metals.
- Process products into samples and submit samples to MEL.
- Complete laboratory analysis of total cadmium and CHCC metals.
- Validate and verify data.
- Enter laboratory data into the PTDB.
- Review QC of data entered into PTDB.
- Submit the data to the CSPA Enforcement Officer.
- Develop the final project report.

4.7 Practical constraints

It may be difficult to obtain proper sample sizes (> 0.25 g) for laboratory testing of individual components of an item of jewelry. When possible, we will purchase small jewelry items in multiples and combine identical jewelry components in order to obtain samples large enough for laboratory testing. After consultation with and approval from the CSPA Enforcement Officer, we will consider alternate processing methods when sample size limitations arise. For example, an earring from a small set of earrings may not produce the required mass for testing when deconstructed into individual components (i.e., decorative adornment, post, and backing). The first and preferred option is to combine the similar components from each of the individual earrings in the set to obtain the required mass. The second option is to combine all the components from the entire set of earrings. The combined earring set will then be submitted as a composite sample and described as such in the PTDB component description. Additional descriptive comments and/or photos will be included as necessary. Composite samples will be limited to similar materials (i.e., metals or plastics). Surface coatings and paints will not be considered as a discernable component. Gemstones will not be evaluated in this study.

4.8 Systematic planning process

This Quality Assurance Project Plan constitutes the systematic planning process.

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Table 2 lists the key individuals involved in this project. All are employees of Ecology.

Table 2. Organization of Project Staff and Responsibilities.

Staff	Title	Responsibilities
Josh Grice HWTR Program Phone: 360-407-6786	Client	Clarifies scope and budget of the project. Provides review of the draft QAPP and approves the final QAPP.
Carol Kraege HWTR Program Phone: 360-407-6965	Client / Section Manager	Reviews and approves the project scope and budget. Provides review of the draft QAPP and approves the final QAPP.
Sara Sekerak Toxic Studies Unit SCS, EAP Phone: 360-407-6997	Project Manager	Researches study area and writes the QAPP. Coordinates with laboratory. Conducts QA review of data, analyzes and interprets data. Writes the draft report and final report.
Christina Wiseman HWTR Program Phone: 360-407-7672	Assistant / Sampling Lead	Procures targeted products. Performs product login, XRF screening and sample preparation.
Dale Norton Toxic Studies Unit SCS, EAP Phone: 360-407-6765	Unit Supervisor for the Project Manager	Provides internal review and approval of the draft QAPP. Reviews and approves the final QAPP.
Will Kendra SCS, EAP 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.
Ken Zarker HWTR Program Phone: 360-407-6724	Section Manager for the Assistant	Reviews the project scope and budget, tracks progress, reviews the draft QAPP, and approves the final QAPP.
Joel Bird Manchester Environmental Laboratory Phone: 360-871-8801	Laboratory Director	Reviews the draft QAPP and approves the final QAPP. Communicates lab activities with Project Manager.
Samuel Iwenofu HWTR Program Phone: 360-407-6346	HWTR Quality Assurance Coordinator	Reviews the draft QAPP and approves the final QAPP.
William R. Kammin Phone: 360-407-6964	Ecology Quality Assurance Officer	Reviews and approves the draft QAPP and the final QAPP.

HWTR: Hazardous Waste and Toxics Reduction
SCS: Statewide Coordination Section
EAP: Environmental Assessment Program
QAPP: Quality Assurance Project Plan

5.2 Special training and certifications

Ecology's published Product Testing Standard Operating Procedure (SOP) will be followed for product selection and documentation, product tracking, and sample preparation (van Bergen, 2014). Ecology staff conducting the XRF analysis will follow the manufacturer's standard operating procedure as defined in the XL3 Analyzer Version 8.0.0 Users Guide (Abridged) Revision A November 2011.

5.3 Organization chart

See Tables 2 and 3.

5.4 Project schedule

Table 3. Proposed Schedule for Completing Product Collection and Laboratory Work, Data Entry into Product Testing Database (PTDB), and Reports.

Field and laboratory work	Due date	Lead staff
Product collection completed	10/2015	Christina Wiseman
XRF screening completed	10/2015	Christina Wiseman
Laboratory analyses completed	11/2015	
Product Testing Database (PTDB) database		
	Due date	Lead staff
Lab data loaded	12/2015	Christina Wiseman
PTDB QA review	1/2016	Sara Sekerak
Data entry complete	2/2016	Christina Wiseman
Final report		
Author lead / Support staff	Sara Sekerak (lead) / Christina Wiseman	
Schedule		
Draft due to supervisor	12/2015	
Draft due to client/peer reviewer	12/2015	
Final (all reviews done) due to publications coordinator	1/2016	
Final report posted to the web	2/2016	

5.5 Limitations on schedule

The schedule must be followed closely to ensure all milestones are met appropriately.

5.6 Budget and funding

The product collection and laboratory costs estimated for this project total $40,500. Table 4 shows the estimated costs for this project.

Table 4. Project Budget and Funding.

Product/ Parameter	Number of Samples	QC Samples*	Cost per Sample	Subtotal	
Product Collection^	250	---	$10	$2,500	---
				Product Collection Total:	$2,500
Cryomilling	40	---	$100	$4,000	---
Metals Analysis	140	30	$200	$34,000	---
				Laboratory Total:	$38,000
				Project Total:	$40,500

*QC samples in this table include those that are not provided free of charge (matrix spikes, duplicates, SRMs and cryomill rinseates).

^ Product collection spending is allocated from a separate budget from project budget.

6.0 Quality Objectives

6.1 Decision Quality Objectives

Decision quality objectives (DQOs) are not necessary for this project.

6.2 Measurement Quality Objectives

Manchester Environmental Laboratory (MEL) is expected to meet all QC requirements for this project. Table 5 lists the specific measurement quality objectives (MQOs), including the lowest concentration of interest. MEL will evaluate all collected data and report any discrepancies to the listed MQOs.

Table 5. Measurement Quality Objectives for Laboratory Analysis.

Analyte	Bias		Precision	Sensitivity
	LCS (% recov.)	Matrix Spikes (% recov.)	Duplicates (RPD)	Lowest Concentration of Interest
Arsenic	85 - 115%	75 - 125%	≤ 20%	1.0 ppm
Antimony	85 - 115%	75 - 125%	≤ 20%	1.0 ppm
Cadmium	85 - 115%	75 - 125%	≤ 20%	1.0 ppm
Cobalt	85 - 115%	75 - 125%	≤ 20%	1.0 ppm
Lead	85 - 115%	75 - 125%	≤ 20%	1.0 ppm
Mercury	85 - 115%	75 - 125%	≤ 20%	0.5 ppm
Molybdenum	85 - 115%	75 - 125%	≤ 20%	1.0 ppm

6.2.1 Targets for Precision, Bias, and Sensitivity

6.2.1.1 Precision

Precision is a measure of the variability in the results of measurements due to random error. Laboratory precision will be assessed through laboratory duplication of product samples. There is no plan to submit field duplicates for this project. See Table 5 for MQOs.

6.2.1.2 Bias

Bias is the difference between the population mean and the true value. Assessments of laboratory bias will be determined by analysis of laboratory control samples (LCSs) and matrix spiked samples. See Table 5 for MQOs.

6.2.1.3 Sensitivity

Sensitivity is a measure of the capability of a method to detect a substance. The lowest concentrations of interest are listed in Table 5.

6.2.2 Targets for Comparability, Representativeness, and Completeness

6.2.2.1 Comparability

Product samples will be purchased, processed and submitted to the laboratory consistent with the procedures described in Ecology's Product Testing SOP (van Bergen, 2014). Adherence to established SOPs, laboratory methods and data verification processes ensure comparability between all product testing projects.

6.2.2.2 Representativeness

Ecology staff will purchase a large number of products (approximately 250) to help ensure that products collected are representative of those available to consumers. Major retailers in the area will be visited to obtain a wide variety of jewelry. Purchasing from Internet sources may also be conducted to obtain product representativeness.

Refer to Tables 1 and 6 for the target numbers of purchased products, distribution and sampling plan.

6.2.2.3 Completeness

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

7.0 Sampling Process Design (Experimental Design)

7.1 Study design

Approximately 250 children's jewelry items will be procured from retailers in the Puget Sound area and through Internet retailers selling to Washington consumers. Products selected based on the purchasing design described in Section 3.1 and illustrated in Table 1 will be brought back to Ecology headquarters, isolated into separate components, and XRF-screened for metals. Component samples will be aided for selection for laboratory analysis based on XRF screenings and are anticipated to be distributed according to the design plan shown in Table 6. Samples containing cadmium will be prioritized for laboratory testing. Samples containing high levels of CHCC metals will be prioritized second and the final samples will be selected across the other remaining metals and follow the sampling design plan.

Table 6. Anticipated Number and Types of Samples Designated for the Analysis of Metals⁺.

Matrix	Jewelry Product Groupings							Total Number of Samples
	Chain or Necklace	Earrings	Armcuff or Bracelet	Rings	Brooches, cufflinks, pins, other ornaments	Body Piercing Jewelry	Hair Accessory, Crown or Tiara	
Metal	20	20	15	15	15	10	5	100
Plastic	10	10	10	10	0	0	0	40

⁺Arsenic, antimony, cadmium, cobalt, lead, mercury, and molybdenum.

7.1.1 Field measurements

Not applicable.

7.1.2 Sampling location and frequency

Children's jewelry will be purchased from a variety of Puget Sound retailers and from on-line retailers over a two-week period in September.

7.1.3 Parameters to be determined

See Table 5 for a list of parameters to be determined.

7.2 Maps or diagram

Not applicable.

7.3 Assumptions underlying design

Cadmium and CHCC metals have been incorporated into children's jewelry.

7.4 Relation to objectives and site characteristics

Not applicable.

7.5 Characteristics of existing data

Ecology's previous studies on chemicals in products were designed to look at a wide range of toxic chemicals and product types. This study will narrow the focus of study to children's jewelry.

8.0 Sampling Procedures

8.1 SOPs

Product collection, screening, and preparation will follow the Product Testing SOP (van Bergen, 2014).

8.2 Containers, preservation methods, holding times

Samples will be stored in 2- or 4-oz. glass jars with no preservation. No storage temperature requirements or holding times have been established for product matrices.

8.3 Invasive species evaluation

Not applicable.

8.4 Equipment decontamination

Equipment decontamination is covered in the Product Testing SOP (van Bergen, 2014).

8.5 Sample ID

Product samples will be labeled with component IDs generated by the Product Testing Database and a sample ID based on the MEL work order. The mass of the sample will be written on the outside of the jar. Specific details of component ID and sample ID generation are described in the Product Testing SOP (van Bergen, 2014).

8.6 Chain-of-custody, if required

A chain of custody will be maintained throughout sample processing, screening, shipment, laboratory analysis, and when necessary, upon return of sample aliquots back to HQ. Ecology staff will use MEL's chain of custody form for samples sent to the laboratory.

8.7 Field log requirements

Photographs, receipts, and store information will be stored in the Product Testing Database upon return from purchasing events. Other documents such as advertisements and webpage information will be stored in the database when applicable.

8.8 Other activities

Product Collection

Staff will record information such as the type of advertisement used to sell the product and the area in the store where the product was found; this will help ensure the product is marketed for children. Staff will take photos at the time of purchase of products and include the adjacent area when there is ambiguity about whether the product is intended for children.

After staff collect all products, they will return to Ecology headquarters and assign a unique product identification number. Additional photos and descriptive notes will be recorded and stored.

Product Isolation

Component isolation of children's products will follow the CSPA Reporting Rule guidelines (Ecology, 2011a). Jewelry will be disassembled into individual components (e.g., earring post, backing, and decorative adornment) and screened by the XRF. Coatings of paint will not be separated as an individual component. Natural gems and stones will be excluded from laboratory testing.

XRF Screening

Metal readings from the XRF will aid in the selection of product components to be forwarded to the laboratory for metals analyses.

The screening by the XRF may be limited due to the small size of jewelry and jewelry components. The XRF requires samples presented for analysis to be of a certain thickness and to cover the analysis window for obtaining accurate results as described in the XL3 Analyzer Version 8.0.0 Users Guide (Abridged) Revision A November 2011. A tin check standard (#180-060, batch H) and polyethylene plastic check standard (lot# T-51), containing the analytes of interest, will be used to verify the performance of the XRF. At a minimum the check standards will be run before and after each product analysis set. The project manager will review the XRF data thoroughly to account for possible error analyses due to sampling (size and/or thickness) and against the performance of the instrument check standards.

Components with XRF measurements indicating the presence of cadmium will be forwarded to the laboratory for analysis. Following cadmium, components with XRF measurements of metals, at or above the screening levels in Table 7, will be considered as a possible laboratory sample. If many components contain target elements above the selection criteria, samples will be prioritized for analysis based on the highest concentrations.

Table 7. XRF Screening Levels for Metals.

Element	XRF Screening Levels (ppm)
Antimony	50
Arsenic	50
Cadmium	20
Cobalt	50
Lead	45
Mercury	NL*
Molybdenum	50

* NL = No limit. Presence of mercury in any concentration constitutes submission of a component for further laboratory analysis.

9.0 Measurement Methods

9.1 Field procedures table/field analysis table

Not applicable.

9.2 Lab procedures table

Table 8. Lab Procedures.

Analyte	Samples (number/ arrival date)	Expected Range of Results	Matrix	RL (ppm)	Cryomill Method	Preparation Method	Analysis Method	Analysis Instrument
Metals*	140, 10/15/2015	< 1.0 - 5000 ppm	Plastic	1 ppm (Hg, 0.5 ppm)	MEL SOP 720033	EPA 3052	EPA 6020A	ICP-MS
			Metal	1 ppm (Hg, 0.5 ppm)	N/A	EPA 3052	EPA 6020A	ICP-MS

*Arsenic, antimony, cadmium, cobalt, lead, mercury, and molybdenum.

9.2.1 Analyte

Samples will be analyzed for the suite of metals of high concern to children: arsenic, antimony cadmium, cobalt, lead, mercury, and molybdenum.

9.2.2 Matrix

The matrices to be analyzed will be limited to metals and plastics.

9.2.3 Number of samples

See Table 8.

9.2.4 Expected range of results

See Table 8.

9.2.5 Analytical method

EPA Method 6020A will be used for the analysis of metals.

9.2.6 Sensitivity/Reporting Limit

See Table 8.

9.3 Sample preparation methods

After XRF screening, staff at Ecology HQ will reduce the product components to approximately 8 mm x 8 mm pieces using stainless steel tools (e.g., wire cutters or snips) before submitting them to MEL for analysis. The final reduced samples will be placed into labeled 2- or 4-oz. jars, and the mass of contained sample will be recorded on the jar. A chain-of-custody will be recorded throughout sample processing, screening, shipment, and laboratory analysis. Detailed product processing procedures are described in Ecology's Product Testing SOP (van Bergen, 2014).

9.4 Special method requirements

MEL SOP 720033 (2014) will be followed for cryomilling the plastic samples. When cryomilling is performed, cryomill rinseate blanks will be collected and analyzed to assess any sample-to-sample carryover during the cryomill process. After each sample is cryomilled, the cryomill grinding jar, grinding ball, and Teflon gasket will be scrubbed with Citranox® five times and rinsed thoroughly with deionized water. The final 50 milliliters of deionized rinse water will be collected and acidified with nitric acid for use as the cryomill rinseate blank; all rinseate blanks will be kept through the end of the project. One rinseate from each batch of twenty samples will be randomly selected and analyzed.

The analyst will assess the rinseate blanks and sample results for the presence of metals and run any additional rinseate blanks as necessary to determine possible carryover.

9.5 Laboratory accredited for methods

MEL will conduct all analyses for metals. MEL is accredited for method EPA 6020A.

10.0 Quality Control Procedures

10.1 Table of field and lab quality control required

Table 9 outlines the laboratory quality control (QC) samples planned for this project. MEL will run cryomill rinseate blanks (when appropriate), method blanks, laboratory control samples (LCS), standard reference materials (SRM), duplicates (DUP), matrix spikes (MS), and matrix spike duplicates (MSD) with each batch of 20 samples. The project manager will designate seven samples to be processed as laboratory duplicates. Low relative percent differences (RPDs) between duplicates evaluated in previous studies indicate that seven duplicates are a reasonable number for this study. MEL will follow all applicable policies, procedures, and SOPs described in the *Manchester Environmental Laboratory Quality Assurance Manual* (MEL, 2012).

Table 9. Quality Control Tests.

Analyte	Cryomill Rinseates	Method Blank	Laboratory Control Sample	Standard Reference Material	Laboratory Duplicate	Matrix Spike	Matrix Spike Duplicate
Metals	1/batch	1/batch	1/batch	1/batch	1/batch	1/batch	1/batch

10.2 Corrective action processes

MEL will document and report any discrepancies to the listed MQOs in Table 5. The project manager shall be promptly notified of issues with sample amounts, cryomilling, blank contamination or sample digestion processes for direction of further recourse. The project manager will determine whether data should be re-analyzed, rejected, or used with appropriate qualification.

If a cryomill rinseate blank identifies cross-contamination as a result of carryover in the cryomill, the affected samples will be qualified following the National Function Guidelines for Inorganic Superfund Data Review (EPA, 2014). Depending on the degree of contamination, the laboratory may be required to reanalyze the affected samples.

11.0 Data Management Procedures

11.1 Data recording/reporting requirements

All project data will be stored in Ecology's Product Testing Database. The database will hold product descriptions, purchase receipts, photos of products, and laboratory data and case narratives. The data will be available to the public through an external search application at: https://fortress.wa.gov/ecy/ptdbpublicreporting/. All data will be reviewed for quality assurance following entry into the database.

11.2 Lab data package requirements

MEL will provide a standard deliverable package after completing their work. All quality control data will be included with the package. MEL will discuss any problems encountered with the analyses, corrective action taken, changes to the requested analytical method, and a glossary for data qualifiers.

The narrative will include:

- Printed reports with QA summaries for all results.
- Explanations of any difficulties encountered during cryomilling, digestion, or analysis.

11.3 Electronic transfer requirements

Case narratives will be in PDF format and electronic data deliverables will be in an Excel spreadsheet format. PDF documents will be sent to the project manager via email and the electronic data deliverable (Excel) will be delivered through the LIMS system.

11.4 Acceptance criteria for existing data

Not applicable.

11.5 EIM/STORET data upload procedures

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

12.0 Audits and Reports

12.1 Number, frequency, type, and schedule of audits

This project will be conducted according to established practices within Ecology which are designed to produce data of acceptable quality and ensure that corrective actions are implemented in a timely manner.

MEL and contracted laboratories must participate in performance and system audits of their routine procedures. No audits are planned specifically for this project.

12.2 Responsible personnel

As per Tables 2 and 3.

12.3 Frequency and distribution of report

A report summarizing findings for this project will be published at the end of the study. The final report will include:

- General descriptions of products purchased.
- Descriptions of product categories.
- Results of laboratory analyses.
- Statistical summaries of laboratory results.
- Summary of laboratory data collected.

12.4 Responsibility for reports

See Tables 2 and 3.

13.0 Data Verification

13.1 Field data verification, requirements, and responsibilities

Not applicable.

13.2 Lab data verification

Qualified laboratory staff will examine laboratory data and document findings in a case narrative. The case narratives will be sent to the project manager as a summation of laboratory data quality. The narrative will include MEL's assurance that the QA Project Plan, methods, and SOPs were followed and all data quality objectives were met. The project manager will review the QC sample results for precision, bias, and accuracy, and verify the quality assurance criteria have been met.

13.3 Validation requirements, if necessary

Independent data validation is not planned for this project.

14.0 Data Quality (Usability) Assessment

14.1 Process for determining whether project objectives have been met

Once the project data has been reviewed and verified, the project manager will evaluate and determine if the study objectives were met. Laboratory QC samples will be reviewed in order to determine if the MQOs were met. Estimates of accuracy and precision will be based laboratory QC. Data will be accepted, accepted with qualifiers, or rejected at the discretion of the project manager.

14.2 Data analysis and presentation methods

The final report will include a statistical summary of the results. Summary statistics, such as minimum, maximum, median, and frequency of detection will be presented in a table.

14.3 Treatment of non-detects

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

14.4 Sampling design evaluation

The number and type of collected samples will be sufficient to meet the objectives of this project.

14.5 Documentation of assessment

Documentation of assessment will occur in the final report.

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16.0 Figures

The figure in this QAPP is inserted after it is first mentioned in the text.

17.0 Tables

The tables in this QAPP are inserted after they are first mentioned in the text.

18.0 Appendix. Acronyms and Glossary

Acronyms and Abbreviations

Following are acronyms and abbreviations used in this report.

CHCC	Chemicals of High Concern to Children
CPSC	Consumer Products Safety Commission
CSPA	Children's Safe Product Act
EAP	Environmental Assessment Program
Ecology	Washington State Department of Ecology
e.g.	For example
EPA	U.S. Environmental Protection Agency
et al.	And others
HWTR	Hazardous Waste and Toxics Reduction Program
i.e.	In other words
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
PBT	persistent, bioaccumulative, and toxic substance
PTDB	Product Testing Database
QA	Quality assurance
QC	Quality control
RCW	Revised Code of Washington
RPD	Relative percent difference
SOP	Standard operating procedures
SRM	Standard reference materials
WAC	Washington Administrative Code
XRF	X-ray fluorescence analyzer

Units of Measurement

ppm	parts per million

Quality Assurance Glossary

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

Accuracy - The degree to which a measured value agrees with the true value of the measured property. USEPA recommends that this term not be used, and that the terms precision and bias be used to convey the information associated with the term accuracy. (USGS, 1998)

Analyte - An element, ion, compound, or chemical moiety (pH, alkalinity) which is to be determined. The definition can be expanded to include organisms, e. g. fecal coliform, Klebsiella, etc. (Kammin, 2010)

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

Blank - A synthetic sample, free of the analyte(s) of interest. For example, in water analysis, pure water is used for the blank. In chemical analysis, a blank is used to estimate the analytical response to all factors other than the analyte in the sample. In general, blanks are used to assess possible contamination or inadvertent introduction of analyte during various stages of the sampling and analytical process. (USGS, 1998)

Calibration - The process of establishing the relationship between the response of a measurement system and the concentration of the parameter being measured. (Ecology, 2004)

Check standard - A substance or reference material obtained from a source independent from the source of the calibration standard; used to assess bias for an analytical method. This is an obsolete term, and its use is highly discouraged. See Calibration Verification Standards, Lab Control Samples (LCS), Certified Reference Materials (CRM), and/or spiked blanks. These are all check standards, but should be referred to by their actual designator. (i. e. CRM, LCS, etc.) (Kammin, 2010; Ecology, 2004))

Comparability - The degree to which different methods, data sets and/or decisions agree or can be represented as similar; a data quality indicator. (USEPA, 1997)

Completeness - The amount of valid data obtained from a project compared to the planned amount. Usually expressed as a percentage. A data quality indicator. (USEPA, 1997)

Continuing Calibration Verification Standard (CCV) - A QC sample analyzed with samples to check for acceptable bias in the measurement system. The CCV is usually a midpoint calibration standard that is re-run at an established frequency during the course of an analytical run. (Kammin, 2010)

Control chart - A graphical representation of quality control results demonstrating the performance of an aspect of a measurement system. (Kammin, 2010; Ecology 2004)

Control limits - Statistical warning and action limits calculated based on control charts. Warning limits are generally set at +/- 2 standard deviations from the mean, action limits at +/- 3 standard deviations from the mean. (Kammin, 2010)

Data Integrity - A qualitative DQI that evaluates the extent to which a dataset contains data that is misrepresented, falsified, or deliberately misleading. (Kammin, 2010)

Data Quality Indicators (DQI) - Data Quality Indicators (DQIs) are commonly used measures of acceptability for environmental data. The principal DQIs are precision, bias, representativeness, comparability, completeness, sensitivity, and integrity. (USEPA, 2006)

Data Quality Objectives (DQO) - Data Quality Objectives are qualitative and quantitative statements derived from systematic planning processes that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions. (USEPA, 2006)

Dataset - A grouping of samples organized by date, time, analyte, etc. (Kammin, 2010)

Data validation - An analyte-specific and sample-specific process that extends the evaluation of data beyond data verification to determine the usability of a specific data set. It involves a detailed examination of the data package, using both professional judgment, and objective criteria, to determine whether the MQOs for precision, bias, and sensitivity have been met. It may also include an assessment of completeness, representativeness, comparability and integrity, as these criteria relate to the usability of the dataset. Ecology considers four key criteria to determine if data validation has actually occurred. These are:

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

Examples of data types commonly validated would be:

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

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

- No qualifier, data is usable for intended purposes
- J (or a J variant), data is estimated, may be usable, may be biased high or low
- REJ, data is rejected, cannot be used for intended purposes (Kammin, 2010; Ecology, 2004)

Data verification - Examination of a dataset for errors or omissions, and assessment of the Data Quality Indicators related to that dataset for compliance with acceptance criteria (MQO's). Verification is a detailed quality review of a dataset. (Ecology, 2004)

Detection limit (limit of detection) - The concentration or amount of an analyte which can be determined to a specified level of certainty to be greater than zero. (Ecology, 2004)

Duplicate samples - Two samples taken from and representative of the same population, and carried through and steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variability of all method activities including sampling and analysis. (USEPA, 1997)

Initial Calibration Verification Standard (ICV) - A QC sample prepared independently of calibration standards and analyzed along with the samples to check for acceptable bias in the measurement system. The ICV is analyzed prior to the analysis of any samples. (Kammin, 2010)

Laboratory Control Sample (LCS) - A sample of known composition prepared using contaminant-free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is prepared and analyzed in the same batch of regular samples using the same sample preparation method, reagents, and analytical methods employed for regular samples. (USEPA, 1997)

Matrix spike - A QC sample prepared by adding a known amount of the target analyte(s) to an aliquot of a sample to check for bias due to interference or matrix effects. (Ecology, 2004)

Measurement Quality Objectives (MQOs) - Performance or acceptance criteria for individual data quality indicators, usually including precision, bias, sensitivity, completeness, comparability, and representativeness. (USEPA, 2006)

Measurement result - A value obtained by performing the procedure described in a method. (Ecology, 2004)

Method - A formalized group of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, data analysis), systematically presented in the order in which they are to be executed. (EPA, 1997)

Method blank - A blank prepared to represent the sample matrix, prepared and analyzed with a batch of samples. A method blank will contain all reagents used in the preparation of a sample, and the same preparation process is used for the method blank and samples. (Ecology, 2004; Kammin, 2010)

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

Percent Relative Standard Deviation (%RSD) - A statistic used to evaluate precision in environmental analysis. It is determined in the following manner:

$$\%RSD = (100 * s)/x$$

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

Parameter - A specified characteristic of a population or sample. Also, an analyte or grouping of analytes. Benzene and nitrate + nitrite are all “parameters” (Kammin, 2010; Ecology, 2004)

Population - The hypothetical set of all possible observations of the type being investigated. (Ecology, 2004)

Precision - The extent of random variability among replicate measurements of the same property; a data quality indicator. (USGS, 1998)

Quality Assurance (QA) - A set of activities designed to establish and document the reliability and usability of measurement data. (Kammin, 2010)

Quality Assurance Project Plan (QAPP) - A document that describes the objectives of a project, and the processes and activities necessary to develop data that will support those objectives. (Kammin, 2010; Ecology, 2004)

Quality Control (QC) - The routine application of measurement and statistical procedures to assess the accuracy of measurement data. (Ecology, 2004)

Relative Percent Difference (RPD) - RPD is commonly used to evaluate precision. The following formula is used:

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

where "Abs()" is absolute value and a and b are results for the two replicate samples. RPD can be used only with 2 values. Percent Relative Standard Deviation is (%RSD) is used if there are results for more than 2 replicate samples (Ecology, 2004).

Replicate samples - Two or more samples taken from the environment at the same time and place, using the same protocols. Replicates are used to estimate the random variability of the material sampled. (USGS, 1998)

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

Sample (field) - A portion of a population (environmental entity) that is measured and assumed to represent the entire population. (USGS, 1998)

Sample (statistical) - A finite part or subset of a statistical population. (USEPA, 1997)

Sensitivity - In general, denotes the rate at which the analytical response (e.g., absorbance, volume, meter reading) varies with the concentration of the parameter being determined. In a specialized sense, it has the same meaning as the detection limit. (Ecology, 2004)

Spiked blank - A specified amount of reagent blank fortified with a known mass of the target analyte(s); usually used to assess the recovery efficiency of the method. (USEPA, 1997)

Spiked sample - A sample prepared by adding a known mass of target analyte(s) to a specified amount of matrix sample for which an independent estimate of target analyte(s) concentration is available. Spiked samples can be used to determine the effect of the matrix on a method's recovery efficiency. (USEPA, 1997)

Split Sample - The term split sample denotes when a discrete sample is further subdivided into portions, usually duplicates. (Kammin, 2010)

Standard Operating Procedure (SOP) - A document which describes in detail a reproducible and repeatable organized activity. (Kammin, 2010)

Systematic planning - A step-wise process which develops a clear description of the goals and objectives of a project, and produces decisions on the type, quantity, and quality of data that will be needed to meet those goals and objectives. The DQO process is a specialized type of systematic planning. (USEPA, 2006)

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