Metal Cookware Leaching Test

Protocol Document

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Contents

Acknowledgements

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Disclaimer

This protocol is designed for use in specific projects and may not be universally applicable. It should be adapted or modified only with the guidance of relevant experts to ensure it meets the unique needs of each project. The creators of this protocol assume no responsibility for its misuse or for any outcomes resulting from its application beyond its intended scope.

Acronyms

AAS	Atomic absorption spectroscopy
EPA	United States Environmental Protection Agency
GPS	Global Positioning System
HBA	Home-based assessment
ICP-MS	Inductively coupled plasma mass spectrometry
RMS	Rapid Market Screening
XRF	X-ray fluorescence analyzer

1. Introduction

The objective of this protocol is to present the step-by-step process to conduct leachate tests for lead in metal cookware.

When a child with an elevated level of lead in their blood (typically >5 µg/dL) is identified, it is important to understand the factors that contribute to the burden of lead in his or her body. Investigating the contributing sources is needed to design an effective intervention for that specific child and his or her family. This also allows for the identification of trends to inform larger interventions at the community-, regional- or even national- level.

Pure Earth's Rapid Market Screening (RMS) program and other supporting research revealed that lead may leach from aluminum cookware into food during cooking, raising the concern of this potential exposure route for children. Other research has shown that brass cookware may also leach high lead levels.

This protocol provides guidance for a standard leaching methodology to evaluate the concentrations of lead potentially leaching from the metal cookpots into food. This method uses acetic acid as a leachate medium to mimic acidic foods, such as tomato sauce. This guidance manual refers the user to the U.S. Food and Drug Administration's (FDA's) "Elemental Analysis Manual (EAM) for Food and Related Products" (Link) for analysis of food products. This document is intended to be used by field and laboratory personnel conducting metal cookware leachate analyses.

2. Sampling Cookware

Please refer to Pure Earth's XRF Cookware Analysis Protocol for instructions on how to select items for analysis, characterize the cookware, conduct XRF analysis, and record data. *Even if the cookware has not been subjected to XRF analysis, it is still important to document the cookware's characteristics according to the methods described in the XRF protocol*.

3. Laboratory Capacity

Ensure the laboratory is qualified to assess heavy metals in water and has experience doing so. Ensure the laboratory will use the specified sample processing and analytical methods (e.g., ICP-MS), and has a well-established Quality Assurance/Quality Control (QA/QC) program (e.g., calibration, sample blanks, duplicates, etc.). Labs should have access to ultra-pure water and ultra-high purity grade reagents. If they do not, the lab should ensure that all necessary quality control procedures are followed, including ensuring that any background concentrations of lead and other metals are thoroughly documented and accounted for in blanks. These labs lab should consider purchasing a large batch of materials to combine for use in their experiments,

which should then be analyzed via ICP-MS to characterize any background levels of metals.

Consider splitting samples for analysis by two labs – and confirm they align with an initial set of samples before analyzing the full set of samples.

4. Leaching Test

4.1 Health and Safety

- The leaching test should be carried out in a laboratory setting following customary lab safety practices, including the use of disposable nitrile (preferred) or latex gloves, eye protection, and protective clothing.

4.2 Test Protocol

- Record the general characteristics of the cookpot using the procedures described in Pure Earth's XRF Cookware Analysis Protocol. Data to be recorded specifically for the leachate test is described below in Section 5.
- Wash the cookpot with a laboratory detergent solution, rinse with deionized water, and allow to air dry.
- Prepare a solution with a 4% volume/volume (v/v) solution of ultra-pure acetic acid[1] and ultra-pure water[2]. Please note that the acetic acid concentration used in this procedure may change, depending on the outcome of further investigations. Please contact Pure Earth for updates.
- Add this acetic acid solution to the cookware up to the 80% capacity fill line.
- Measure the pH of the solution with a calibrated pH meter.
- Cover the cookware with its lid (preferred, if available) or silicone sheet to reduce evaporation, then:
    - If using cookware with a flat bottom, place cookware on an electric hot plate.
    - If using cookware with a rounded bottom, place cookware on a heating mantle such as these to ensure stability and uniform heating. In the absence of a mantle, place the cookpot on a flat pan of sand on top of an electric hot plate.

[1] If ultra-pure acetic acid is not available, then reagent grade may be used if the background concentrations of metals in the acetic acid is determined.
[2] If ultrapure water is not available, then deionized or distilled water may be used. In this event, it is important to determine the background concentrations of metals in the water.

- Bring the acetic acid solution in the cookware to a simmer, noting the amount of time it took for the solution to come to the desired temperature.
- If the acetic acid solution does not come to a full simmer, hold cookware at the maximum temperature achieved for the two-hour period. Record the achieved temperature.
- Check the volume of liquid in the cookware against the fill line every 15 minutes. To compensate for evaporation, add additional acetic acid solution to the fill line as needed.
    - After 2 hours (while hot), take a 100 mL sample of the simmering solution using a glass pipette and place in a high-density polyethylene (HDPE) container[3].
    - After 2 hours of simmering, turn off the heat and allow the acetic acid solution to remain at room temperature for 24 hours. Measure the pH of the solution with a calibrated pH meter and collect another 100 mL sample

4.3 Cooling and Preservation

- Note: If possible, sample containers should be refrigerated overnight until analysis.
- As the cookware cools, make note of:
    - The presence or absence of precipitate in the cookware.
    - The presence or absence of precipitate in the sample container.
    - The color of any precipitate (e.g., black, gray, tan).
- To preserve the sample, add sufficient nitric acid (HNO_3) to achieve a pH of <2. This may be done at the time of sample collection or after transportation to the lab.

4.4 Quality Assurance and Quality Control

- Blanks, spikes, and duplicates should be carried throughout the process to confirm the accuracy of the test. At a minimum, this should include:
    - Blank: Conduct the leachate test using a cookware sample with a lead concentration of 0 ppm.
    - Spike: Add a known quantity of lead to the sample prior to conducting the leachate test.

[3] LDPE containers may be appropriate, if they are of high enough quality to prevent leakage during storage.

- Duplicate: Conduct the leachate test on two identical samples with identical concentrations of lead.

4.5 Preparation for Analysis

4.5.1 Health and Safety

- Concentrated nitric and hydrochloric acids can irritate the skin and mucous membranes. If available, use a fume hood when handling these reagents. The safety protocol should also include the use of gloves, eye protection, and protective clothing.

4.5.2 Digestion

If precipitate is present, ensure that the entire sample is digested to completion.

4.5.3 Analysis

- Samples should be analyzed using Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) according to US EPA SW-846 Method 6020.
- An appropriate Pb reporting limit is 1 ppb, but specificity is relative to your laboratory's detection limits.
- **Request a copy of QA/QC procedures and results from the analytical lab.**

5. Data Recording

This section is designed to record data from the leachate experiments. Please complete as many of the following data fields as possible in the provided template:

Variable	**Data entry**	**Definition**
Investigator	Free-form	Full name of investigator
OrganizationTesting	Free-form	Organization conducting the analysis

Variable	Data entry	Definition
Cookpot_ID	Free-form	From Cookpot ID generated in General Dictionary
Date	Format enforced	Date leachate testing initiated
Time	Format enforced	Time leachate testing initiated
AceticAcid%	Free-form	Acetic acid concentration (in percent)
Initial_pH	Free-form	Initial pH of acetic acid at room temperature
AchievesSimmer	Dropdown	Did the acetic acid come to a simmer? Yes/No
AceticTemp	Free-form	If the acetic acid did not come to a simmer, temperature achieved (in Celsius/Centigrade). Otherwise, NA
InitialSimmerTime	Free-form	Simmer (or heating if no simmer) time before first sample (hours)
SecondSimmerTime	Free-form	Simmer (or heating if no simmer) time before second sample (hours)
RT_Time	Free-form	Time at room temperature before final sample (hours)
Final_pH	Free-form	Final pH of acetic acid after cooling to room temperature
Initial_PbConc	Free-form	Analytical Pb result after initial simmering (in micrograms per milliliter - ug/ml)
Second_PbConc	Free-form	Analytical Pb result after second simmering (in micrograms per milliliter - ug/ml)

Variable	**Data entry**	**Definition**
RT_PbConc	Free-form	Analytical Pb result after resting at room temperature (in micrograms per milliliter - ug/ml)
Precipitate	Dropdown	Was a precipitate noted in the sample? Yes/No
Notes	Free-Form	Additional narrative
Definitions: *Dropdown* fields restrict data entry to defined choices from a dropdown menu *Format enforced* fields restrict data entry to specific formats *Free-form* means that the analyst can enter unstructured text, without specific formatting		