

#6

Proposal Coversheet

# Application Submitted

To: Dr. Arlene Courtney  
Program: Ch 462 Experimental Chemistry  
Title of Project: Analysis of Lead Leaching from Ceramic Tableware  
Suggested Timeline: One week  
Date Submitted: January 26, 2009  
Principle Investigator  
Applicant's Contact Information

## Abstract

Fiestaware is a highly collectable line of ceramic tableware with bright colored glazes. In order to achieve these bright colors, uranium oxides were used; however, uranium is radioactive and decays to a stable isotope of lead. Previous research has found that the lead can leach into acidic solutions where it can then enter food. This proposal outlines why lead leaching is a health risk, examines previous research, and proposes a method for the analysis of leached lead in an acidic solution from a bowl that Western Oregon University owns. This analysis will determine the concentration of lead using an atomic absorption spectrophotometer and the results will be checked against the FDA's accepted levels of lead in leach solution from ceramicware.

date: 1/26/09

## Introduction

During the 1930's, many pottery companies began producing inexpensive ceramic tableware.<sup>1</sup> Until the 1960's, many of the glazes used contained elevated levels of radioactive materials, including uranium, thorium, and/or potassium-40.<sup>2</sup> Because these compounds are radioactive, they are unstable and emit alpha, beta, and gamma particles through a decay series until a stable isotope is reached.<sup>1,2</sup>

Homer Laughlin China Company was one company that began producing this tableware.<sup>1</sup> In 1936, they began producing a line of ceramic tableware called Fiestaware.<sup>3</sup> This highly collectable line of ceramics featured bright, bold colors and art deco styling.<sup>1,3</sup> In order to create the vibrant orange-red colors for the glaze, uranium oxides were used in the glaze.<sup>2</sup>

It has been estimated that each Fiestaware plate may contain up to 14.5 grams (g) of uranium. For the glaze used on Fiestaware, uranium-238 (U-238) was used causing the ceramicware to be radioactive. The decay series of U-238 ends in the stable isotope of lead-206 (Pb-206). Therefore not only was Fiestaware glazed with a radioactive element, but it also contains lead that can leach from the glaze in an acidic solution.<sup>1</sup> The ability of the lead to leach into an acidic solution is of great concern.

Lead that is leached into an acidic solution can enter into humans through consumption of food prepared on ceramics containing lead in the glaze. Adverse health effects of lead poisoning can be seen with as little as 4 micrograms per milliliter ( $\mu\text{g/mL}$ ) in the blood.<sup>4</sup> Lead is usually accumulated gradually in the body and may not be noticed until irreversible damages have appeared. Lead poisoning can have many signs and symptoms. Children are more susceptible to lead poisoning and may show symptoms such as weight loss, abdominal pain, vomiting, paleness, and learning difficulties. Lead is also dangerous to adults, and symptoms can include

pain, numbness, and tingling in extremities; headaches; abdominal pain; memory loss; mood disorders; and low sperm count or abnormal sperm.<sup>5</sup>

Many federal agencies have set levels of lead that are allowed to be present in different mediums, such as air, water, and food. In addition, the Food and Drug Administration (FDA) has set levels that are allowed to be present in leach solutions from ceramicware. As of 2008, the FDA has stated that 0.5 to 3.0  $\mu\text{g}/\text{mL}$  can be present in the leaching solution.<sup>4</sup> Specifically, the FDA has set the following levels: pitchers and cups/mugs at 0.5  $\mu\text{g}/\text{mL}$ ; large hollowware other than pitchers at 1.0  $\mu\text{g}/\text{mL}$ ; small hollowware other than cups/mugs at 2.0  $\mu\text{g}/\text{mL}$ ; and flatware at 3.0  $\mu\text{g}/\text{mL}$ .<sup>6</sup>

The Chemistry department at Western Oregon University (WOU) possesses several dishes that may contain lead in the glaze. The bowls were produced during the time the manufacturer was known to use uranium oxide-based glazes. The purpose of this study is to determine if the bowl contains lead and how much is leached into an acidic solution. Once these levels are known, the safety concerns of these bowls can be determined.

### **Justification**

Previous research has shown that lead leaching continues to be a problem. The problem of lead leaching from glazes has been known for over a century; however, in some parts of the world it continues to be a concern. According to Weinhold, researchers followed 321 children in Mexico from 1987 to 1992. During this time, the use of lead as an additive to gasoline was banned. However, the researchers found that many of the children still had higher blood levels of lead. Many of these children came from families that used ceramics with a lead based glaze. These children had blood lead concentrations 18.5% higher than in children whose families did not use ceramics with lead based glazes.<sup>7</sup>

Although efforts have been made by the United States government to reduce lead exposure by banning lead-based paint, addition to gasoline and use in food canning, lead exposure is still of great concern. Research by Lynch et al, found that in a Hispanic community located in Oklahoma City had higher blood lead levels (BLLs) due to their regular use of ceramic cooking ware containing lead-based glazes. They tested 25 ceramic vessels in the community and found that 52% contained lead levels higher than the FDA's accepted levels. Many of the ceramic cookware they tested had been in the family for many years or brought to the United States from Mexico where warnings regarding use with food may be absent.<sup>8</sup>

Somogyi et al did research with terracotta pottery in Hungary, where it is common to store solid and liquid foods in these containers; many of which contain a lead-based glaze. In each of the 11 pieces of pottery, tea, tea with lemon, and vinegar were stored for varying amounts of time and then tested for lead. Based on their data, it was observed that the amount of lead leached from the pottery increased with increasing storage time and depended on the item being stored in the pottery.<sup>9</sup>

This previous research also found that informing the families of the glaze on the ceramics did not help much as the families still used them on occasion or children used them in other family member's homes.<sup>7</sup> Additionally, some families used ceramics and other pottery that has been brought to the United States from other countries where lead levels are not regulated at all.<sup>8</sup> In addition the research by Somogyi et al showed that the resistance of the glaze depended on the manufacturing process and improper firing of the glaze can lead to increase lead leaching.<sup>9</sup> According to Wienhold, the researchers found that while regulations on lead levels are in place in Mexico, the businesses producing the ceramics are poorly monitored and many of the businesses are small family-owned companies with no quality control.<sup>7</sup> Even if the lead levels

are monitored and quality controls are in place, lead can still be leached into food through use of poorly manufactured ceramics or those made in small family-owned operations.

## **Materials and Methods**

Previous research has used various methods, such as inductively coupled plasma (ICP) and energy dispersive X-ray fluorescence (EDXRF) spectrometer, to determine the lead concentration in a leaching solution.<sup>8,9</sup> However, WOU does not have access to the instruments for these methods. Many of the previous researchers cited the Official Methods of Analysis of AOAC International as a reference. The following method is adapted from the Official Methods of Analysis of AOAC International Lead and Cadmium Extracted from Ceramicware method.<sup>10</sup> This method utilizes an atomic absorption spectrophotometer (AAS), which WOU does have., specifically, a Buck Scientific 200A AAS will be used.

**Apparatus** The AAS will be equipped with a light source specific to lead. An air-acetylene flame will be used. The absorbance of lead will be measured at 217.0 and 283.3 nm, depending on the lead concentration. Additional flasks, beakers, and stirring rods will be needed throughout the procedure.

**Reagents** A 4% acetic acid solution will be used to leach lead from the glaze. This solution can be prepared by diluting glacial acetic acid with water or can be purchased commercially. Because we are interested in the concentration of lead leached during personal use of the Fiestaware, table vinegar (typically 4 to 8% concentration) will be used. Because the actual concentration of acetic acid in the vinegar is not necessary, it will not be determined. The vinegar will be used in all stock and standard solutions as well.

To determine the lead concentration that leaches from the ceramicware, solutions of lead nitrate ( $\text{PbNO}_3$ ) of known concentrations will be prepared. A stock solution will be prepared by

dissolving 1.5985 g of  $\text{PbNO}_3$  in vinegar and diluting to 1 L with vinegar. Solutions of increasing lead concentration will be prepared by diluting 0.0, 1.0, 2.0, 3.0, 5.0, and 10.0 mL of the stock solution to 1L with vinegar, creating standard solutions with concentrations of 0, 1, 2, 3, 5, 10  $\mu\text{g/mL}$ .

**Extraction** The Fiestaware bowl should be washed with lukewarm water and a detergent used to wash dishes, rinsed with tap water followed by distilled water and allowed to dry. The bowl will then be filled with vinegar to within 6-7 mm of overflowing. The bowl should be covered to prevent evaporation, but the cover should not come into contact with the solution. The vinegar will stand in the bowl for 24 hours at  $22 \pm 2$  °C. After 24 hours the level of vinegar will be checked. If the volume has decreased, additional vinegar will be added to adjust volume to the start volume. The solution will be stirred and a sample removed by pipetting into a clean container.

**Analysis** The absorbance of the lead standard solutions will be determined first. A sample of vinegar will be aspirated, the AAS will be zeroed, and then the  $\text{PbNO}_3$  standard solution will be aspirated. Each standard will be aspirated three times, and zeroed between each sample. The three absorbances will be averaged. Based on the absorbances and known concentrations, a calibration curve will be created using Microsoft Excel.

The sample of the leach solution will be analyzed on the AAS in the same manner as the standard solutions. The AAS will be zeroed using vinegar, and then the leach solution will be aspirated. This will be done three times and the absorbances averaged. Based on the calibration curve, the concentration of lead in the leach solution will be determined. This concentration will be compared to the levels allowed by the FDA to determine health risks with this piece of ceramic tableware.

## Budget and Timeline

**Budget** This analysis is estimated to cost very little. WOU laboratory already possess the AAS for performing the analysis. In addition, the laboratory already has the beakers, flasks, and glass stirring rods that will be needed. Because this experiment is also a learning opportunity for the students in the chemistry program, WOU will provide the necessary reagents. However, the reagents can easily be purchased from the grocery store and chemical supply companies. Vinegar and dish detergent can be purchased for approximately \$10. In addition, 500 g lab grade lead nitrate can be purchased from Fisher Scientific for \$12.75.

**Timeline** This analysis will take approximately a week to complete. On Monday, February 9<sup>th</sup>, the bowl will be filled with the vinegar. The next day, the leach solution will be checked, and additional vinegar added if need. A sample from the bowl will be removed. On Monday, February 16<sup>th</sup>, the standard and leach solution will be analyzed by the AAS and calculations completed.

## Sources

- (1) Lead Analysis Proposal. <http://www.wou.edu/las/physci//ch462/proposal.htm> (accessed January 16, 2009).
- (2) Radioactive Materials in Antiques. <http://www.epa.gov/rpdweb01/antiques.html> (accessed January 16, 2009).
- (3) The Homer Laughlin China Co. <http://www.hlchina.com/index.htm> (accessed January 15, 2009).
- (4) ATSDR – ToxFAQs – CABS – Lead. <http://www.atsdr.cdc.gov/cabs/lead/index.html> (accessed January 16, 2009).
- (5) Lead Poisoning – MayoClinic.com. <http://www.mayoclinic.com/health/lead-poisoning/FL00068> (accessed January 15, 2009)
- (6) Sec. 545.450 Pottery (Ceramics); Imported and Domestic – Lead Contamination (CPG 7117.07). [http://www.fda.gov/ora/compliance\\_ref/cpg/cpgfod/cpg545-450.html](http://www.fda.gov/ora/compliance_ref/cpg/cpgfod/cpg545-450.html) (accessed January 18, 2009).
- (7) Weinhold, B. Lead in Mexican Children: Pottery Uses Slow Reductions in Blood. *Environmental Health Perspectives*. **2004**, 112 (10), A 569.
- (8) Lynch, R.; Elledge, B.; Peters, C. An Assessment of Lead Leachability from Lead-Glazed Ceramic Cooking Vessels. *Journal of Environmental Health*. **2008**, 70, 36-40.