

INTRODUCTION

Lead poisoning awareness has increased dramatically over the past couple decades. However, most concerns lie in lead based paint used around the house or workplace. Another source of dangerous lead levels is in dinnerware, more specifically, Mexican pottery that has a lead-based glaze on it. These dishes are no longer sold in the United States for this reason, but that does not negate the fact that they are still in existence. The lead can become leachable for a number of reasons including poor firing in the manufacturing process, cracks or chips in the ceramic ware, or even regular use in the microwave or dishwasher. The Food and Drug Administration has set a compliance guideline of $\leq 3 \mu\text{g/mL}$ of lead in a leaching agent.¹ What is to be determined is the actual lead content in these dishes.

JUSTIFICATION

In order to determine if the dishes are dangerous, the lead content is needed. A quick experiment can be done to determine this. Measuring how much lead leaches out of the bowls into an acid should help determine the lead content. The time and materials required are minimal, but the results will be applicable to families everywhere especially considering the cases already recorded.

LITERATURE REVIEW

Studies have already been done to determine if the levels of lead leaching off dinnerware could be harmful, and the FDA has even set up specific guidelines for testing ceramic dishes for their lead content. A study by the San Diego Department of Health Services released in 1997 measured the blood lead levels in a Hispanic community.² After testing ceramic pottery used for eating out of, 31% was found to be positive for lead. Further testing of beans cooked in the ceramic ware revealed lead levels ranging from 0.7-35.9 $\mu\text{g/g}$. Another study done in New York City was released in the Morbidity and Mortality Weekly Report in 2004 outlining the monitoring of a single patient showing high blood lead levels.¹ The patient was an infant whose blood lead levels continued to rise, despite close observation and testing of their surroundings. It was eventually determined that the elevated lead levels were caused by commercially manufactured ceramic dinnerware. The testing was done following protocol outlined by the FDA. The protocol is outlined in the FDA Elemental Analysis Manual for Food and Related Products.³ The protocol describes what acid to use, the instruments and materials needed, the analysis method, how to calibrate the instrument, and the calculations needed to determine the relevance of your findings.

METHOD

Summary of Method

Lead can easily be extracted from the dinnerware by applying an acidic solution to the surface. Once the lead has leached into the acid, the lead content can be measured using Atomic Absorption Spectroscopy. A calibration curve is to be obtained for the sample to be compared to.

Materials

To perform this method, beakers, flasks, and stirring rods will be used, preferably made of chemically resistant plastic, borosilicate glass, quartz, or Teflon. The acid is to be 4.0% acetic acid, and a lead stock solution of 1000 $\mu\text{g/mL}$ is also needed. If the lead stock solution is not available it can be prepared by dissolving 1.5985 g $\text{Pb}(\text{NO}_3)_2$ in 4.0% acetic acid and diluted to 1.0 L. Standard lead solutions can then be made from the stock solution by diluting 0.0, 1.0, 2.0, 3.0, 5.0, and 10.0 mL of the stock solution to 1 L with acetic acid. A lead check solution should also be obtained, either as another bottle of stock or made from another source of $\text{Pb}(\text{NO}_3)_2$.⁴

Sample Preparation

The simplest way to prepare the sample is to fill a ceramic bowl with acetic acid (vinegar) and allow it to sit for 24 hours at 22 degrees Celsius. The bowl should be covered to prevent evaporation and the initial level marked so if evaporation occurs it can be adjusted. After 24 hours check the level to see if any evaporation has occurred, if it has, add acetic acid to replenish and carefully stir the solution. If a precipitate is present, a PTFE filter may be used to remove it. The samples should be placed in sealed containers, and the analysis should be done by the following day.^{3,4}

Instrument Setup

A screening test should be done first on the leach solution using flame atomic absorption spectroscopy. The target range for the instrument is 0.050 to 0.400 Abs. If the initial measurement does not fall into the target range, it can be diluted with more vinegar. The concentration can then be found using the Abs, taking into account any diluting performed. If the tested leach solution measured an Abs less than or equal to the 0.050 minimum, flame AAS cannot be used and graphite furnace AAS is required. Since this was just a screening process, the initial measurements are not valid until the instrument has been calibrated. The instrument should be set with the following parameters: wavelength of 217.0 nm, Pb lamp, background correction, with the air and C_2H_2 flow rates set according to the manufacturer.^{3,4}

Sample Measurement

Determine the Abs of the standard lead solutions and produce a calibration curve with a linear equation. Run leach samples and compare Abs to calibration curve to determine lead content. The burner should be flushed with water in between each test. The Abs of the check solution also needs to be determined and the measured concentration must be within 5% of the known level.⁴

BUDGET AND TIMELINE

The materials needed to perform the analysis are relatively inexpensive. Acetic acid in the form of common vinegar is readily available, and the glassware needed is common to any laboratory. The largest expense lies in the instrumentation, but since the laboratory has an atomic absorption spectrometer, the only cost lies in operation. Costliness is not an issue for this analysis. The time required to perform this analysis is also minimal. The preparation needed should be no more than 5-10 minutes, and then after it has set for 24 hours, it should take no more than a couple hours to complete the analysis.

1. Galvez, M; Vanable, L; Forman, JA; Landrigan, PJ; Akeredolu, E; Leighton, J; Nagin, D. Childhood lead poisoning from commercially manufactured French ceramic dinnerware. *MMWR* **2004**, 53, 584-585.
2. Gersberg, Richard M.; Gaynor, Kate; Tenczar, Donald; Bartzen, Martha; Ginsberg, Michelle; Gresham, Louise S.; Molgaard, Craig. Quantitative modeling of lead exposure from glazed ceramic pottery in childhood lead poisoning cases. *IJEHR* **1997**, 7, 193-202.
3. *FDA Elemental Analysis Manual*; Food and Related Products, U.S. Food and Drug Administration, CFSAN, Maryland, 2000; 4.1.

4. Lead and Cadmium Extracted from Ceramicware. In *Official Methods of Analysis of AOAC*, 16; Cunniff, Patricia A.; AOAC International: Gaithersburg, MD, 1998; 1, chapter 9, pp 6-6a.