filtrates & residues

edited by
JAMES O. SCHRECK
University of Northern Colorado
Greeley, CO 80639

Testing for Lead in the Environment

Karen A. Sundback
Department of Physical Sciences
San Jose City College
San Jose, CA 95128

This experiment is directed towards students in their first chemistry course for two reasons. First, the required laboratory techniques and knowledge of chemistry are minimal. And second, it answers the question that arises most frequently in this first course, which is, "Why are we learning this; what good is chemistry?" The main purpose of this experiment is to test quantitatively for lead in household items. Recently, there has been much concern in the news media regarding the presence of lead in the environment. Lead is an acknowledged health risk and, as a result, has been phased out of most manufacturing and processing in the United States in an attempt to eliminate it from our daily lives; nonetheless lead still remains a threat:

- Prior to 1950, most paints contained lead. In 1978, lead was eliminated from paints. However, you may still be exposed to lead if you live or work in an old building whose paint is peeling. Peeling paint can form a dust that may be inhaled into the lungs.
- Glazes used on ceramics currently manufactured in the United States should not contain lead. Nevertheless, beware of ceramics from other countries or older ceramics in your home.
- Lead is sometimes used in paints on plastic bags and is still used in lead crystal.
- Although lead was phased out of gasoline by 1991, you may want to think twice before planting your vegetable garden next to a freeway. (The test described here is *not* sensitive enough to detect lead in soils near freeways nor in water supplies having pipes with lead solder.)

The problem is that, despite being phased out, lead is still present. It is the major source of heavy metal poisoning and probably the biggest environmental threat to young people: 4 million American children have been poisoned by lead. Ingested or inhaled lead enters the bloodstream and inhibits the production of hemoglobin, which carries oxygen throughout the body. It also inactivates enzymes in the brain and nervous system. Depending upon the concentration of lead, symptoms of lead poisoning may vary from flulike symptoms to personality changes in adults and learning disabilities in children to, at the highest concentrations, death.

Reactions Involved in the Experiment

This particular test is sensitive to 20 ppm or as little as 0.1 mg of Pb^{2+} in the final 5 mL of water. Although this procedure is not sensitive enough to test for lead in water, it certainly can detect lead in paints and glazes. In testing an item for lead, the lead must first be leached from the item. This can done by adding acetic acid; the soluble and colorless lead(II) acetate will result.

$$Pb^{2+}(s) + 2HC_2H_3O_2(l) \rightarrow Pb(C_2H_3O_2)_2(aq) + 2H^+(aq)$$

The acid must then be evaporated to prevent it from catalyzing a reaction in which potassium iodide is oxidized by oxygen to elemental iodine. The resulting yellow iodine solution would give a false positive result; that is, it would indicate lead even when lead is not present.

Once the acid is evaporated and the item has cooled, potassium iodide is added and if lead is present, a yellow precipitate will form.

$$\underset{\text{colorless}}{Pb(C_2H_3O_2)_2(aq)} + 2KI(s) \rightarrow \underset{\text{yellow precipitate}}{PbI_2(s)} + 2KC_2H_3O_2(aq)$$

Procedure

• **Caution:** Wear safety goggles throughout the experiment!

Experimental note: Students should bring items to be tested from their homes. Household items most likely to contain lead include ceramic mugs, house paints from before 1978, paint on plastic bags, and lead crystal glass. However, forgetful students may obtain samples from lecture or laboratory room wall paint or paint on pencils.

Reagents: Concentrated acetic acid ($HC_2H_3O_2$), Potassium iodide KI, crystals.

Equipment: Oven placed in hood, oven tongs, 10-mL graduated cylinder, 30-mL beakers. It is important to clean all glassware and equipment, using deionized (DI) or distilled water in the final rinse.

Sample Preparation

Ceramics, such as coffee mugs and beer steins

Add 25 mL of acetic acid to ceramic item, swirl, and place in oven to evaporate the acid. Continue by following *General Procedure.*

Crystal glass

Add 25 mL of acetic acid to item; swirl. To protect item in oven, put it into a large beaker and place in oven to evaporate the acid. **Lead crystal can crack if it cooled too quickly;** thus, after the acid has evaporated, turn the oven off and let the crystal cool in the oven with the door open at least 15 min. Continue by following *General Procedure.*

House paints

To collect paint chips, use a knife to scrape paint from an inconspicuous area. The total amount of paint chips collected should be larger than the size of a thumbnail. Add paint chips and 25 mL of acetic acid to a clean 30-mL beaker. Evaporate the acid by placing the beaker in the oven. Continue by following *General Procedure.*

Paint on plastic bags

Cut all painted areas from bag and pack these into clean 30-mL beaker using clean stirring rod. Add 25 mL of acetic acid to beaker, using stirring rod to push plastic bag into acid and to dissolve paint from bag. When most

of the paint is dissolved, use stirring rod to carefully remove plastic bag to another beaker for disposal. (Wash off the acetic acid before disposing of this bag. One way to do this is to fill and empty the beaker several times with water. When acetic acid is washed off, dispose of plastic bag into trash can.) Evaporate acid from paint by placing in oven. Continue by following *General Procedure.*

General Procedure

1. When all samples are in the oven, the instructor or a student will turn the oven on, making sure the final oven temperature reads 150–200 °C. Close the hood doors if possible. After an hour, the instructor should check for complete evaporation of the acid by first checking that the color of the litmus paper is blue (see instructor notes). If so, approach the hood and, if there is no odor of acid at all, open the hood doors carefully. If any odor of acid escapes, close doors immediately. If no odor of acetic acid remains in the hood, open the oven door carefully. If any odor of acid escapes, close the door immediately and raise the oven temperature to 175–200 °C.

• **Caution:** Avoid inhaling acetic acid fumes.

2. When evaporation is complete, turn oven off and remove sample with oven tongs (except crystal; allow crystal to cool in oven), keeping tongs on outside surface of sample.

3. Let sample cool a few minutes (crystal should be cool to the touch). Then add 5 mL of DI water; swirl it around the inside of item. This solution should be clear and colorless. *For ceramic items,* pour this water into a beaker labeled "KI" to make sure that this water is colorless; then pour the water back into ceramic sample.

4. To develop color, add a pea-sized amount of KI crystals to the DI water and swirl it to dissolve. If lead is present a yellow precipitate should form immediately. *For ceramic items,* to check for yellow precipitate, pour this KI solution into "KI" beaker.

Instructor's Notes

In our tests, no paint on pencils has tested positive for lead. Nevertheless, it's an interesting test.

Litmus paper can be used to determine when the acetic acid has been driven off. Tape blue litmus paper onto a clean conspicuous surface near the oven vent.

Test reagents for lead contamination by running a blank sample, and provide students with an example of a positive result by running a control sample. To do this, clean two small beakers, rinse with deionized water, and, fill both with 25 mL of concentrated acetic acid. To the control sample add at least 1 mg of elemental lead. Place beakers in oven to evaporate acid. When acetic acid has evaporated to dryness, remove beakers from oven, and when cooled, fill each with 5 mL of DI water. Add a pea-sized amount of KI crystals. Blank sample should contain no lead. Any formation of color would be due to contamination of reagents or residual acetic acid. Control sample has lead; addition of KI should result in yellow precipitate.

There is no evidence that the presence of other ions will either prevent the formation of the lead iodide precipitate or mask its brilliant yellow color.

Disposal

Students should discard the final KI solution according to directions given by the laboratory instructor.

Conclusions

This experiment usually generates interest from even the most apathetic students because it relates to their lives: lead is usually found in a number of the ceramic mugs; indeed, in our trials we found lead in the paint peeling from laboratory and lecture room walls.

Other advantages of this experiment include the fact that a minimal amount of toxic chemicals are used and generated. Also, the experiment is easy for most students and can be scheduled in conjunction with other activities such as quizzes or study sessions.

Literature Cited

1. *Indoor Air Pollution;* U.S. House of Representatives. Committee on Energy and Commerce. Subcommittee on Health and the Environment. U.S. Government Printing Office: Washington, DC, 1991.
2. *Lead Poisoning;* U.S. House of Representatives. Committee on Energy and Commerce. Subcommittee on Health and the Environment. U.S. Government Printing Office: Washington, DC, 1991.
3. *Lead-Based Paint Hazard in American Housing;* U.S. Senate. Committee on Banking, Housing, and Urban Affairs. Subcommittee on Housing and Urban Affairs. U.S. Government Printing Office: Washington, DC, 1992.
4. *The Lead Exposure Reduction Act of 1993;* U.S. Senate. Committee on Environment and Public Works. Subcommittee on Toxic Substances, Research, and Development. U.S. Government Printing Office: Washington, DC, 1993.
5. *Lead in Ceramicware and Crystal;* U.S. Senate. Committee on Governmental Affairs. Ad hoc Subcommittee on Consumer and Environmental Affairs. U.S. Government Printing Office: Washington, DC, 1992.

Homeowner's Test for Lead in the Paint

Thomas Rees
Phillips Academy
Andover, MA 01810[1]

This test will tell if there is lead in paint. It will not tell how much lead is present.

Scrape a sample of the paint, about the size of a nickel or a dime, from the wood. Be sure that the scraping gets down to the wood at the bottom of the paint, so that you will get all of the layers of the paint.

Put a stink bomb, really a glass ampoule, in a jar with a good screw top, like an old peanut butter jar. Screw the top tightly and shake the jar until the ampoule is broken. Stink bombs can be purchased at a novelty store.

Do the test outdoors. Open the jar, add the paint sample quickly, and close the jar. Tilt the jar so that the liquid from the stink bomb will wet the paint. If there is lead in the paint, the surface will turn black.

The stink bomb contains ammonium sulfide. The black color is lead sulfide. If the paint contains only titanium dioxide, the black color will not appear. If the paint sample is old lead paint covered with lead-free paint, only one side of the sample will be black. The other side will remain white.

It may seem more sensible to add the paint sample to the jar at the same time as the stink bomb. But shaking the jar pulverizes the paint, and the test is less visible. Adding the paint sample later allows only a trace of the stink to escape, and you have a more visible test of the lead sulfide.

[1]Present address: 35 Sunset Rd., Hamden, CT 06514