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**Science Education Title** Lead Analysis of Soil Using Atomic Absorption Spectrometry (AAS)

**Overview**

Atomic absorption spectrometry is based on the absorption of discrete wavelengths of light by ground-state, gas-phase free atoms. Atoms of different elements absorb characteristic wavelengths of light. A hollow cathode lamp (fig 1) is used to emit light with the specific frequency that can be absorbed. The energy absorbed excites the electrons in the target element from their ground-state to a higher-energy state. The amount of light absorbed is proportional to the concentration of the element in the sample. Using a standard curve, the concentration of the element in the sample can be determined.

AAS is an elemental analysis technique that can provide quantitative information on over 70 different elements. Concentrations as low as ppt (parts per trillion) can be determined for some elements, with ppb (parts per billion) and ppm (parts per million) being more common for various metals. This method has several benefits over others. For example, this technique measures the total concentration of an element, regardless of its form. In addition, the wavelength used is specific to the element being tested, so there will be no interference from other elements in the sample, making it a fast and easy technique.

**Principles**

Lead occurs naturally in soil in levels ranging from 50 – 400 ppm. However, with the widespread use of lead in paint and gasoline, in addition to contamination by industry, urban soils often have concentrations of lead significantly greater than background levels – in some places up to 10,000 ppm. Lead does not biodegrade, but remains in the soil.

Serious health risks are associated with lead poisoning. Children are particularly at risk, and exposure can lead to developmental and behavioral problems for them. The Environmental Protection Agency has set a standard for lead in soil at 400 ppm for play areas and 1200 ppm for non-play areas.

Lead in soil is also of concern when it is soil that is to be used for gardening. Plants take up lead from the soil. Therefore, if one eats vegetables or herbs grown in contaminated soil, lead can be ingested. Contaminated soil particles can also be breathed in while gardening or brought into the house on clothing or footwear. It is recommended that soils with lead levels greater than 400 ppm not be used for gardening. It is further recommended that soil with lead levels between 100 and 400 ppm not be used for leafy vegetables or herbs due to the fact that lead can be stored in the leaves. Root vegetables should also not be grown in this soil due to the fact that lead can accumulate in plant roots as well.

**Procedure**

**1. Soil Preparation**

1.1. Mix sample thoroughly and sieve using a USS #10 sieve.

1.2. Dry soil in an oven at 40°C for 24 hours.

**2. Sample Digestion**

2.1. Using an analytical balance, weigh out 1g of soil sample and place in a digestion tube. Record the weight of the sample to four decimal places.

2.2. In a hood, add 5 mL of water to the digestion tube.

2.3. Add 5 mL of concentrated HNO3 to the digestion tube.

2.4. Mix the slurry with a stirring rod. Cover the digestion tube with a tear drop glass stopper.

2.5. Put the digestion tube in the block digester and heat the sample to 95°C and reflux for 10 minutes without boiling. REMEMBER- THIS CONTAINS CONCENTRATED ACID.

2.6. Allow the tubes to cool to room temperature. Add 5 mL of concentrated HNO3 to the digestion tube, replace the drop glass and reflux for an additional 30 minutes. If brown fumes are generated, repeat this step over and over until no brown fumes are given off by the sample.

2.7. Evaporate the solution to a 5 mL volume without boiling.

2.8. Allow the tubes to cool. Add 2 mL of distilled water and 3 mL of 30% H2O2. Cover with the glass stopper and heat to begin the peroxide reaction. Be careful that the solution does not boil over. Heat until the bubbling stops and allow to cool.

2.9. Continue to add 30% H2O2 in 1 mL increments with warming until the bubbling is minimal. Do not add more than a total of 10 mL of the 30% H2O2.

2.10. Cover the sample with the glass teardrop stoppers and heat until the volume is reduced to 5 mL without boiling.

2.11. Add 10 mL concentrated HCl to the sample and cover with the glass teardrop stopper. Heat to 95°C and reflux for 15 minutes.

2.12. Allow the tubes to cool. Filter the sample if there are particulates using filter paper (or similar) and collect filtrate in a 100 mL volumetric flask. Dilute the sample volume to 100mL with distilled water.

**3. Analyzing Samples on Atomic Absorption Spectrometer**

3.1. Turn on the computer and the spectrometer.

3.2. Set parameters on instrument:

3.2.1 Acetylene pressure > 700 kPa (~100 psi)

3.2.2 Acetylene valve set to 11 psi

3.2.3 Air valve set to 45 psi

3.3 Open the SpectraAA software.

3.4. Open a new worksheet in the software.

3.5. Choose “Add Method” and click on Pb to do a Lead Analysis.

3.6. Set Type/Mode parameters:

3.6.1 Type = Flame

3.6.2 Element = Pb

3.6.3 Sampling Mode = Manual

3.6.4 Instrument Mode = Absorbance

3.6.5 Flame Type = Air/Acetylene

3.6.6 Air Flow = 13.5

3.6.7 Acetylene Flow = 2.0

3.6.8 Online Diluter Type = SIPS

3.7. Set the Measurements parameters:

3.7.1 Measurement Mode = PROMT

3.7.2 Calibration Mode = Concentration

3.7.3 Times: Measurement = 10

3.7.4 Times: Read Delay = 10

3.7.5 Replicates: Standard = 3

3.7.6 Replicates: Sample = 3

3.7.8 Precision (%): Standard = 1.0

3.7.9 Precision (%): Sample = 1.0

3.8. Set the Optical parameters:

3.8.1 Lamp Position = Ca #4

3.8.2Lamp Current (mA) = 10.0 mA

3.8.3 Wavelength = 422.7 nm

3.8.4 Slit = 0.5 nm

3.8.5 Background = BC Off

3.9. Set the SIPS parameters:

3.9.1 Nebulizer Uptake Rate = 5.0 mL/min

3.9.2 Right Pump = none

3.9.3 Standard Additions = Unselect

3.9.4 Calibration Mode = Auto Set Std Concentrations

3.9.5 Dual Pump Calibration = Unselect

3.10. Under the Standards tab, a list of standards will automatically populate for the particular test.

3.11. Exit the Edit Method menu and click on the “Labels” tab. Input information regarding sample names and number of samples.

3.12. On the “Analysis” tab, use the “Select” button to highlight the samples to be analyzed.

3.13. Turn on the flame by pressing the ignite button on the instrument.

3.14. Zero the instrument by aspirating a blank and pressing the “Alt” and “Read” keys simultaneously.

3.15. Place the pump tubing in the blank solution and press “Start.” Once the calibration has been performed, place the pump tubing in the sample and press the “Read” key. Continue for all samples.

3.16. Turn off the instrument by pressing the red power off button on the instrument. Turn off all gas tanks and remove all samples.

**Representative Results**

The software will create the calibration curve (figure 2) and automatically determine the concentration of the Pb in the samples.

The values given on the worksheet are mg/L of Pb in the sample *solution*. Additional calculations must be done to convert this number to the ppm of Pb in the *soil* sample.

Example:

For a soil sample that weighed 1.2523 g before digestion and was measured by the AAS to have 6.0 mg/L of Pb in the 100 mL solution sample.

100 mL solution x 6.0 mg Pb x 1 L = 0.6 mg Pb   
 L solution 1000 mL

0.6 mg Pb x 1000 g = 479 mg Pb = 479 ppm  
1.2523 g soil 1 kg kg soil

**Applications**

Atomic Absorption Spectrometry is a useful technique to analyze a wide-range of environmental samples (e.g. water, soil, sludge, sediment) for a large number of elements (e.g. heavy metals). This experiment highlights the use of flame AAS to determine the Pb content in soil (table 1). However, it could also be used to measure concentrations of Cu, Fe, Mn, K, Na, Mg, and Zn in soils.

Atomic Absorption Spectrometry can also be used on non-environmental samples, including:

* Water analysis (Ca, Mg, Fe, Al, Ba, Cr)
* Food analysis (Cd, Pb, Al, Cu, Fe)
* Additives in oils (Ba, Ca, Na, Li, Zn, Mg, V, Pb, Sb)
* Fertilizers (K, B, Mo)
* Clinical samples (blood, serum, plasma, urine, Ca, Mg, Li, Na, K, Fe, Cu, Zn, Au, Pb)
* Cosmetics (Pb)
* Mining (Au)

Legend:  
Figure 1: Hollow cathode lamps from an atomic absorption spectrometer

Figure 2: Calibration curve