

CHEM 4114: Determination of Heavy Metals by Atomic Absorption Spectroscopy (AAS)

Objectives: To determine the concentration of metal ions in a dirt sample using flame atomic-absorption spectroscopy (AAS).

Reference: Principles of Instrumental Analysis, 5th Edition, Skoog, Holler, and Nieman, Chapters 1 (standard addition, page 16 -18), Chapter 6, and Chapter 8.

Instrumentation: Graphite Furnace atomic-absorption spectrometer.

Chemicals: 6M HCl, conc. HNO₃, 2M HNO₃, deionized (DI) water, Pb standard solutions, dried soil.

WARNINGS: Use extreme caution when working with concentrated acids and perform the soil digestion in a fume hood.

Procedures:

Week 1 of the laboratory:

Soil Digestion:

Sample Preparation: Using a standard EPA method (following pages), digest the dirt sample to make a solution of metal ions.

Standard Preparation:

Prepare standard solutions of Pb by dilution of the 1000 ppm stock solution. From these standards, determine the LOD (limit of detection), the LOQ (limit of Quantitation), and the linear dynamic range of the measurement.

Week 2 of the laboratory:

Continue/finish the standard working curve preparation. Determine the amount of Pb in the digested Dirt sample based on the working curve.

Once the amount of Pb has been determined by comparison to the working curve, use the method of standard addition to determine the amount of Pb in the sample.

Instrument Operation:

Install and align an appropriate hollow-cathode lamp in the spectrometer. (See Chapter 5 in the Hardware Guide. Press the Energy button to get a readout of the lamp energy through the monochromator.)

Measurements:

- On the graphite furnace AA instrument, measure the signal of a blank, the standards (from lowest concentration to highest), and the unknown. (Record 3 sets of 5 replicates for each sample).

Data Treatment:

1. Plot working curves for Pb.
2. Determine the concentrations of the Pb in solution. Convert to concentration in the dirt and report the mean and 95% confidence interval.
3. Report the LOD, LOQ, and linear dynamic range of the measurement.
4. Comment on the accuracy of using a calibration curve vs. the method of standard addition for quantitation.