

EXPERIMENT 9

THE DETERMINATION OF Pb IN BRASS BY ATOMIC ABSORPTION SPECTROSCOPY

A. Prelab

- Come to lab prepared. Read Skoog et al. sections 27M-1 (pp. 768-769) for background information.
- Perform the experimental portion of the lab in groups of 2-3. Divide the work so everyone participates.
- You have two lab periods to complete the experimental portion of the lab. Everyone is responsible for completing and handing in a Lab Report.

B. Experimental Overview

In this experiment you will determine the concentration of Pb in a sample of brass. Brass can contain approximately 1-20 wt-% Pb as well as Cu, Zn, and Sn. Atomic absorption spectroscopy (AAS) allows rapid determination of the concentration of Pb in a dissolved sample. While the accuracy is not as great as that obtainable with gravimetric or volumetric techniques, the time required for the determination is considerably less.

C. Procedure

Prepare a 100 ppm stock standard solution

- Weigh to the nearest 0.0001 g approximately 0.08 g of dry, reagent grade $\text{Pb}(\text{NO}_3)_2$ (mw=331.20 g/mol). Dissolve in a solution of 5 mL water and 1-3 mL concentrated HNO_3 . Dilute to 500 mL in a volumetric flask and mix well.

Prepare working standards

- Prepare a set of working standards containing 0, 5, 10, 15, 20 ppm Pb by pipetting the appropriate volume of 100 ppm stock standard solution into a 100 mL volumetric flask, add 5 mL of concentrated HNO_3 and 5 mL of concentrated HCl and dilute to the line.

Prepare triplicate dissolved samples

- Obtain an unknown brass sample from the instructor. Weigh to the nearest 0.0001 g ~0.2 g of each sample into 250 mL beaker. Dissolve the sample by gently boiling the sample with a mixture of 5 mL of concentrated HNO_3 and 5 mL of concentrated HCl while covered with a watch glass. Allow the solution to cool and quantitatively transfer to a 250-mL volumetric flask, dilute to 250 mL, and mix well. Transfer 20 mL of your sample to a 100 mL volumetric flask, add 5 mL of concentrated HNO_3 and 5 mL of concentrated HCl and dilute to the line.

Determine Pb concentrations via AAS

- Set the monochromator of the spectrometer to 283.3 nm, and measure the absorbance of your blank, standards, and samples. Take at least three readings for each measurement. After you have made measurements for your 3 unknowns, measure absorbances for your highest concentration standard. Treat this data as a QC check.

Calculations

- Calculate the concentration of your stock Pb standard.
- Calculate the concentrations of your working standards.
- Calculate a linear regression for your calibration data..
- Determine the concentration of Pb in your diluted sample and calculate the mean and standard deviation wt.-% Pb in your unknown.
- Calculate the relative error of your measurement of the QC sample compared to its actual concentration.
- Calculate a final result \pm CL (95%). Use your calculated s in this calculation.

D. Lab Report

- Title Page (include lab title, your name, your section, and unknown #)
- Purpose (be brief, 1 paragraph)
- Data (Tabular format)
- Calibration curve plot (plot and label your triplicate analyses and your QC check on this graph).
- Calculations (show representative calculation of each type requested above)
- Result

This report will be due one week after completion.