SC306 Instrumental Analysis

Atomic Absorption and Atomic Emission Spectroscopy

Introduction.

Atomic absorption and atomic emission spectroscopy are very useful techniques for detecting trace amounts of metals in a wide variety of sample matrices. Atomic absorption (AA) spectroscopy involves a measurement of the decrease in intensity of a light beam as it passes through a sample. The sample must be in the form of a vapor that contains dissociated atoms. These criteria are met by subjecting the sample to the highly energetic conditions (approximately 3000 C) of a flame or an electrically heated furnace. AA measurements are highly specific since electronic transition energies are unique for each element. However, since atomic spectral lines are quite narrow (around 0.005 nm), the light sources used in this technique must produce well-defined spectral lines. Typically hollow cathode lamps, in which metal atoms, that were dislodged from the cathode surface and excited, emit radiation as they return to the ground state. This emitted light is sufficiently narrow to be absorbed by the metal atoms of interest in the sample. Generally, a different, unique lamp is needed for the analysis of each metal of interest in the sample therefore; simultaneous detection of metals is not practical by this spectral technique. Typically, detection limits are in the parts-per-billion (ppb) or parts-per-million (ppm) range for AA methods.

In atomic emission (AE) spectroscopy, the sample is subjected to even higher temperatures than are used in AA methods (up to 6000 C when an inductively coupled plasma (ICP) source is employed). This high energy environment converts the aqueous sample into a vapor that contains dissociated atoms. Furthermore, the atoms are in excited states. As the excited atoms decay to lower energy states, some of the energy is emitted as light. The intensity of this emitted light is measured at various wavelengths to identify and quantify the metal atoms present in the sample. The high temperature sources employed in AE act to populate a large number of different energy levels for several different elements simultaneously. Therefore, all of the excited atoms can emit their characteristic radiation at the same time and the detector can measure the emission at several different wavelengths (characteristic of several different metals) concurrently. Typically, detection limits are in the ppm range for AE methods.

Procedure: In this experiment, the amount of copper in a brass sample will be quantified using AA and AE and the presence of other metals (such as zinc, tin, or lead) will be confirmed using AE.

Copper Standard Preparation:

1. IN THE HOOD, place about 0.5 g of copper (record the actual weight that you use) into a 250 mL beaker and slowly add 15 mL of AA-grade concentrated nitric acid. Cover the beaker with a watch glass until the evolution of gases ceases.
2. Dilute (by adding the acidic solution to deionized water) this solution to a total volume of 500 mL in a volumetric flask. Calculate its concentration in ppm and label the flask appropriately. This is your copper stock solution.
3. From this stock solution, prepare a series of standard solutions (50 mL each) in the range of 1 to 20 ppm for the ICP-AE analysis and in the range of 1 to 20 ppb of for the AA analysis.

Brass Sample preparation:

1. IN THE HOOD, place about 0.5 g of a brass sample into a 250 mL beaker and slowly add 15 mL of AA-grade concentrated nitric acid. Cover the beaker with a watch glass until the evolution of gases ceases.

2. Dilute (by adding the acidic solution to deionized water) this solution to a total volume of 500 mL in a volumetric flask. Calculate its concentration in ppm and label the flask appropriately.

3. Using the approximate copper content of the brass sample, prepare two 50 mL samples for analysis. One of these samples should fall into the range of your AA copper standards and the other into the range of your AE standards.

Instrument Operation and Data Manipulation.

After your instructor verifies that your standard and sample solutions are within the appropriate concentration ranges for each instrument, he/she will demonstrate the operation of each of these expensive instruments. You will then use your standard solutions to construct an AA and an AE calibration curve for copper and use them to quantify the copper in your brass sample. Report the %Cu in the original brass sample. You will also verify the presence of tin, lead, or zinc in your brass sample using AE (however, you will not quantify these metals).

Questions.

What does the AA-grade label on the nitric acid mean? Why did you use this more expensive grade of acid in this experiment?

What is the main instrumental difference, with respect to detection, between the AA and the AE?

List the different detection wavelengths used in the AA and AE portions of this experiment. Why did you choose these particular wavelengths (remember to mention possible interfering elements)?

Why are the detection limits for AA and AE so different?

References:

Skoog, D.A., Leary, J. J. *Principles of Instrumental Analysis, Fourth Edition;* Harcourt Brace: New York, 1992; chp. 10 and 11.

Fredeen, K. J., Boss, C. B. *Concepts, Instrumentation, and Techniques in Inductively Coupled Plasma Atomic Emission Spectrometry*; Perkin-Elmer Corporation, 1989; chp. 1 and 2.