Graphite-Furnace Atomic Absorption Spectrophotometry

I. Introduction

Atomic absorption spectrophotometry (AAS) has become a routine method for the determination of many trace elements in a variety of sample matrices. One of the limitations of flame AAS is the sensitivity of the method. However, the sensitivity of detection for most elements is significantly improved using a graphite furnace in place of the flame to atomize the sample. In this approach, the liquid sample is placed on a small platform or in a small cup in the furnace and heated in a series of progammed steps to dry, pyrolyze and ultimately atomize the sample. Frequently, a matrix modifier is added to the sample prior to heating so that the chemical form of the analyte is controlled during the heating sequence. Although solid samples can also be analyzed with the furnace, it is generally more desirable to dissolve such samples to minimize matrix effects. In this experiment, a sample of wheat flour will be wet ashed to dissolve the material and the resulting solution containing ppb of Mn will be analyzed using graphite furnace AAS.

II. Equipment

A. Perkin Elmer 3300 AA spectrophotometer with HGA-600 graphite furnace
B. wet ashing kit (obtain from instructor)
C. plastic sampling vials
D. three 50-mL volumetric flasks
E. four 100-mL volumetric flasks

III. Reagents

A. Manganese standard solution, 100 ppm
B. Magnesium nitrate matrix modifier solution, 0.0100 g/mL
C. conc. H_2SO_4
D. 30% H_2O_2

IV. Procedure

A. Wet Ashing of Wheat Flour Sample

1. Accurately weigh out about 0.2 g of the wheat flour and quantitatively transfer it to a 25-mL round bottom flask.

2. Add 2-3 mL of conc. H_2SO_4 to completely wet the sample. Heat the flask with sample in the sand bath for about 15 minutes using a Variac setting of 40.

3. Turn off the heat but leave the flask with sample in the sand bath. Slowly add 1-2 mL 30% H_2O_2 dropwise to completely dissolve the blackened sample.

4. When the sample has dissolved completely (a clear, light yellow solution remains), quantitatively transfer the solution to a 50-mL volumetric flask and dilute to the mark with deionized water.

B. Preparation of Standard Solutions

1. Carefully prepare a 100 ppb solution of Mn by succesive dilutions of the 100 ppm Mn standard solution. Do not use less than 1.0 mL samples for these dilutions.

2. Using volumetric flasks, prepare 30, 50 and 70 ppb Mn standard solutions by the appropriate dilution of the 100 ppb Mn standard solution.

C. Furnace AAS of Mn Standards and Flour Sample

Your instructor will demonstrate the basic use of the software used for data collection and printing. Take careful note of all settings.

1. Prepare a series of plastic sampling vials (6 total) by filling each to the line with either one of the Mn standard solutions, deionized water, matrix modifier solution or the wheat flour sample solution.

2. Place the sampling vials in the autosampler and note the location of each. Your instructor will demonstrate how to program the analysis method with this location information. For each run, use 5 µL of sample (either deionized water for blank, Mn standard or wheat flour solution), 10 µL of diluent (deionized water) and 5 µL of matrix modifier solution.

3. Replace the autosampler cover and put the autosampler in STANDBY mode. Manually manipulate the robotic sampling tube to ensure that it is properly aligned to take up and deliver samples. Remove from STANDBY.

4. Run each sample through the default Mn heating cycle and print the results of each run. Verify that the net peak areas for the three standards bracket that for the unknown.

5. When finished, remove all samples from the autosampler.

V. Treatment of Data

A. Prepare a calibration curve using the net peak area data for the three Mn standards and the 0,0 point. Fit these data points with a linear least-squares line. Print this graph with the equation for the line.

B. Calculate the concentration of Mn in the dilute wheat flour solution from the equation for the calibration curve and the net peak area of the unknown.

C. Calculate the concentration (in ppm) of Mn in the solid wheat flour sample. Report this result.

Lab Cautions for Graphite-Furnace AAS

1. **Before turning on the instrument, ensure that the argon gas and cooling water are turned on.**

2. **Before running any samples, ensure that the autosampler is properly aligned to deliver sample to the furnace platform.**

3. **After placing sampling vials in the autosampler, make sure the lid is properly aligned so that the robotic sampling tube can access the liquid in the sampling vials.**

4. **The last user of the day should turn off the power to the main unit and the furnace power supply, then turn off the argon gas and the cooling water. The computer monitor should also be turned off. Do not exit the software completely; exit to the Desktop before quitting.**

5. **When finished, remove all sampling vials from the autosampler and dispose of the soiutions.**