Atomic Absorption Spectroscopy

I. Introduction

The determination of calcium and magnesium in water samples, blood serum or plasma is of great importance in maintaining clear water supplies and in diagnosing many pathological conditions such as kidney disease, hyperparathyroidism and diabetes. Before the advent of the atomic absorption (AA) spectrophotometer, methods of analysis were tedious and mostly unsatisfactory, usually involving titration with EDTA using poor quality indicators.

The AA method allows calcium and magnesium to be determined quickly and reliably. The preparation of the sample is usually simple and rapid, and concentrations of calcium and magnesium ion at the parts per million level (ppm) are easily measured. Interference by counter ions or complexing ions is minimal, except for phosphate ion suppression of calcium, which can be countered by addition of lanthanum (III) ion to the solutions to be measured. For calcium, the absorption is measured at a wavelength of 422.7 nm, and magnesium absorption is measured at a wavelength of 285.2 nm.

II. Procedure

A. Stock Solutions

Calcium ion (1000 ppm)
Magnesium ion (12.5 ppm)

B. Intermediate Solutions (make all dilutions using class A glassware)

Calcium ion (50.0 ppm)
Dilute 5.00 mL of the stock solution to exactly 100 mL with deionized water.

Magnesium ion (1.25 ppm)
Dilute 10.00 mL of the stock solution to exactly 100 mL with deionized water.

C. Test Solutions

1. Blank solution: deionized water.

2. Prepare calibration standards of Ca^{2+} and Mg^{2+} in 100-mL volumetric flasks as follows:

Std 1: 5.00 ppm Ca^{2+} + 0.0625 ppm Mg^{2+}
10.00 mL Ca^{2+} intermediate solution
5.00 mL Mg^{2+} intermediate solution
Dilute with deionized water to the mark and mix well.

Std 2: 10.00 ppm Ca^{2+} + 0.1250 ppm Mg^{2+}
20.00 mL Ca^{2+} intermediate solution
10.00 mL Mg^{2+} intermediate solution
Dilute with deionized water to the mark and mix well.

Std 3: 12.50 ppm Ca^{2+} + 0.1875 ppm Mg^{2+}
25.00 mL Ca^{2+} intermediate solution
15.00 mL Mg^{2+} intermediate solution
Dilute with deionized water to the mark and mix well.

Std 4: 15.00 ppm Ca^{2+} + 0.2500 ppm Mg^{2+}
30.00 mL Ca^{2+} intermediate solution
20.00 mL Mg^{2+} intermediate solution
Dilute with deionized water to the mark and mix well.

D. Aqueous Unknowns

1. Prepare an unknown sample for Ca^{2+} and Mg^{2+} by the following procedure. Pipet 10.00 mL of the unknown sample into a 100-mL volumetric flask, dilute to the mark and mix well.

2. In addition to the unknown sample, you are encouraged to analyze the tap water at your home along with a sample of water that has been purified if you use any purification or water softening system. Consult with your instructor about collecting such samples.

E. Experimental Measurements Using the AA Spectrophotometer

The lab instructor will demonstrate the operation of the AA spectrophotometer and discuss the instrument settings for this analysis. Carefully note all settings used for your measurements.

1. Measure the absorbance for Ca^{2+} and Mg^{2+} in each standard in order to prepare a calibration plot of standard absorbance versus concentration (in ppm) for each analyte. Measure the absorbance of the unknown solution(s) under the same conditions as those used in determining the standard curves for calcium and magnesium.

III. Calculations

1. Plot the magnesium data (include 0,0 as a data point) and fit with a linear least-squares line. Record the slope and intercept of the line. Plot the calcium data (include 0,0 as a data point) and fit with a second-order polynomial (quadratic) least-squares line. Record the equation of the line. Print a copy of each calibration curve.

2. From the equation of the line for each calibration curve, determine the concentration of Ca^{2+} and Mg^{2+} in the diluted unknown solution(s). Calculate and report the concentration (in ppm) of Ca^{2+} and Mg^{2+} in your original unknown solution(s). **MAKE SURE YOU TAKE INTO ACCOUNT THE SAMPLE DILUTION FACTOR WHEN YOU CALCULATE THE FINAL RESULTS.**