

# 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 renal and liver 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 absorbance is measured at a wavelength of 422.7 nm, and magnesium absorbance is measured at a wavelength of 285.2 nm.

## II. Procedure

### A. Stock Solutions

Calcium ion (1000 ppm)  
Magnesium ion (50.0 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 (5.00 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  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  in 100-mL volumetric flasks as follows:

**Std 1:** 5.00 ppm  $\text{Ca}^{2+}$  + 0.250 ppm  $\text{Mg}^{2+}$   
10.00 mL  $\text{Ca}^{2+}$  intermediate solution  
5.00 mL  $\text{Mg}^{2+}$  intermediate solution  
Dilute with deionized water to the mark and mix well.

**Std 2:** 10.00 ppm Ca<sup>2+</sup> + 0.500 ppm Mg<sup>2+</sup>  
20.00 mL Ca<sup>2+</sup> intermediate solution  
10.00 mL Mg<sup>2+</sup> intermediate solution  
Dilute with deionized water to the mark and mix well.

**Std 3:** 12.50 ppm Ca<sup>2+</sup> + 0.750 ppm Mg<sup>2+</sup>  
25.00 mL Ca<sup>2+</sup> intermediate solution  
15.00 mL Mg<sup>2+</sup> intermediate solution  
Dilute with deionized water to the mark and mix well.

**Std 4:** 15.00 ppm Ca<sup>2+</sup> + 1.000 ppm Mg<sup>2+</sup>  
30.00 mL Ca<sup>2+</sup> intermediate solution  
20.00 mL Mg<sup>2+</sup> intermediate solution  
Dilute with deionized water to the mark and mix well.

### 3. Aqueous Unknown

Prepare an unknown sample for Ca<sup>2+</sup> and Mg<sup>2+</sup> 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.

### D. 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. You may wish to consult the methods manual for additional details.

1. Measure the absorbance for Ca<sup>2+</sup> and Mg<sup>2+</sup> 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 under the same conditions as those used in determining the standard curves for calcium and magnesium.
2. Fit the magnesium data with a linear least-squares line and record the slope and intercept of the line. Fit the calcium data with a second-order polynomial (quadratic) least-squares line and record the equation of the line. Print a copy of each calibration curve.
3. From the equations for the calibration curve least-squares lines, determine the concentration of Ca<sup>2+</sup> and Mg<sup>2+</sup> in the diluted unknown solution. Calculate and report the concentration (in ppm) of Ca<sup>2+</sup> and Mg<sup>2+</sup> in your original unknown solution. **MAKE SURE YOU TAKE INTO ACCOUNT THE SAMPLE DILUTION FACTOR WHEN YOU CALCULATE THE FINAL RESULTS.**