

The Determination of Calcium and Magnesium in Blood Serum and Urine

Application Note

Atomic Absorption

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Introduction

Since the publication of the first papers describing the application of atomic absorption spectrophotometry to the analysis of clinical materials by Willis, [1–4], in 1960, the technique has gained rapid and wide acceptance. Although atomic absorption is eminently suitable for the determination of trace metals, possibly the greatest impact has been on the determination of calcium and magnesium in clinical and biological materials, [5–13].



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Determination of Calcium

In the determination of calcium in both blood serum and urine various diluents have been employed for the suppression of interferences, due to the presence of protein and phosphate.

Blood serum samples are generally diluted 1:10 to 1:20-fold with aqueous solutions of strontium chloride, lanthanum chloride or EDTA, prior to aspiration into a fuel-rich air-acetylene flame.

Urine samples are generally diluted 1:50 to 1:100-fold prior to aspiration into the flame.

Some workers have pointed out that in order to obtain very accurate results the blood and urine samples should first be de-proteinised by precipitating protein with trichloroacetic acid and then diluting with either a solution of lanthanum chloride or strontium chloride, in order to eliminate phosphate interference.

Determination of Magnesium

The determination of magnesium has been carried out on samples of serum and urine that have simply been diluted 1:20 to 1:50-fold with distilled water prior to aspiration into an air-acetylene flame. However, the results of other workers have shown that there is a small protein interference effect. Some workers have preferred to precipitate the protein with 10% trichloroacetic acid, whilst others have simply controlled the protein effect by using diluents which contained strontium chloride, EDTA, lanthanum chloride, or hydrochloric acid.

A description is given here of a method that has been successfully employed at Agilent Technologies for the analysis of both calcium and magnesium in blood serum and in urine.

Experimental

Instrumentation

A Techtron Model 1000 atomic absorption spectrophotometer was used.

	Calcium	Magnesium
Resonance Line (Å)	4226.7	2852.1
Photomultiplier	R-213	R-213
Lamp Current	4 mA	3 mA
Spectral Band Pass (Å)	2	2
Flame	N ₂ O-C ₂ H ₂	Air-C ₂ H ₂
Burner	AB-50, 5 cm	AB-51, 10 cm

Sample Solutions

0.5 mL of blood serum is diluted and made up to 25 mL in a volumetric flask with an aqueous solution of lanthanum chloride. The diluted serum solution should contain 5000 ppm La (strontium chloride may also be used instead of lanthanum chloride). Similarly, 0.5 mL urine is diluted to 50 mL.

Depending on the concentration of calcium and magnesium in the sample, other dilution factors may have to be employed.

In cases where only very small volumes of blood serum are available, 0.1 mL of sample may be diluted to 5 mL or 10 mL. However, great care must be exercised in pipetting out such small sample volumes, if a high degree of precision and accuracy of the results is required.

Standard Solutions

Composite standard solutions are prepared which contain:

1.0 ppm Ca + 0.2 ppm Mg

2.0 ppm Ca + 0.4 ppm Mg

3.0 ppm Ca + 0.6 ppm Mg

Each standard solution should contain 5000 ppm lanthanum (or strontium).

An aqueous solution containing 5000 ppm La is used as a blank.

Results and Discussion

The use of the nitrous oxide-acetylene flame is recommended for the determination of calcium at the 4226.7 Å resonance line. The lanthanum present in the solutions will control not only any interference effect but will also act as an ionization suppressant.

If nitrous oxide is not available then an air-acetylene flame may be employed. However, it should be borne in mind that in the latter flame the sensitivity is approximately 3X less so that this has to be taken into account when preparing standard solutions and also when choosing suitable sample dilution factors.

Magnesium is determined in the air-acetylene flame at the 2852.1 Å resonance line. It is also possible to use the hotter nitrous oxide-acetylene flame, as long as a suitable ionization suppressant is present in both sample and standard solutions (for example, La, Sr, K, Na).

When it is required to operate over a wider concentration range than that given by the above standards it is necessary to prepare higher concentration standards and to use burner rotation in order to reduce the sensitivity of the atomic absorption measurements.

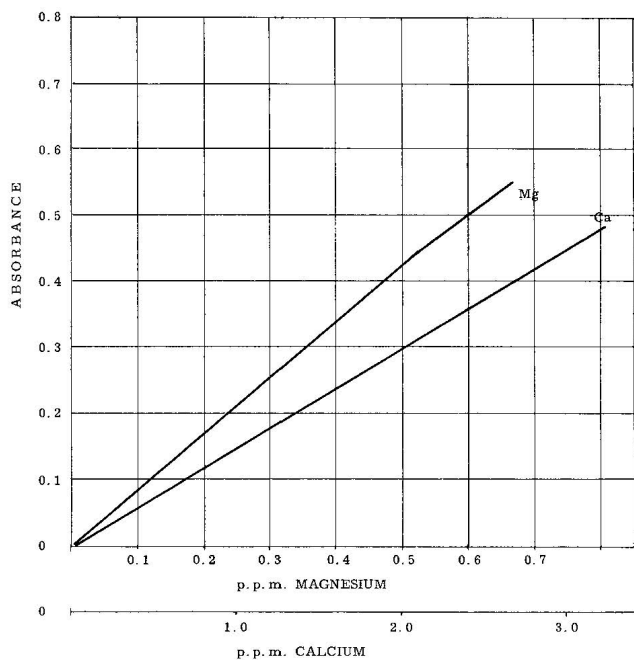


Figure 1. Calibration graphs for calcium and magnesium.

Conclusion

The routine analysis of calcium and magnesium in both blood serum and urine by atomic absorption spectrophotometry is extremely simple and rapid, and yields accurate results.

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