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Application News

Spectrophotometric Analysis No.A408

# Ca Analysis by Atomic Absorption

Ca exists in the natural world in various compounds such as carbonates in the form of marble and limestone, etc., in gypsum ($CaSO_4$), fluorites ($CaF_2$), and apatites ($Ca_5(PO_4)_3F$), etc. Ca forms silvery white-colored crystals and reacts at ambient temperature with oxygen and halogens. It also bonds with oxygen, carbon, nitrogen, silicon and phosphorus when heated. Calcium forms divalent compounds, many of which are white in color. Calcium has a wide variety of applications, such as in bleach and as a pool water treatment agent in the form of calcium hypochlorite (Ca(OCl)Cl), and in stomach antacid preparations in the form of $CaCO_3$ and $Ca(OH)_2$, etc.

A substantial portion of the human body consists of calcium, comprising about 1 kg of a 70 kg adult, 99% of which is contained in hydroxy apatite in the bones, and in muscle at levels of 140 - 700 ppm, and in blood at about 61 mg/dL. Ca performs various critical roles in the body, in particular, as the primary structural component of bone, maintaining the stability and permeability of biological membranes, and for the contraction of muscles, etc. For example, it is essential that sufficient calcium and phosphate be provided for bone formation, and that the Ca concentration in blood plasma is maintained at about 10 mg/dL. The daily intake of Ca in an adult is 600 - 1400 mg, but if the Ca concentration in blood decreases, the muscles shrink and exhibit spasms due to neural excitation. In cases of Ca deficiency, $CaCl_2$ is given either orally or via intravenous injection. On the other hand, hypercalcemia can cause kidney calcification attended by uremia. Since the deficiency and excess of Ca can exert such a great influence in the human body, nowadays, various supplements and functional foods that maintain the Ca balance in the body are being developed and marketed. This Application News presents a type of interference that may occur in flame analysis of Ca, and an effective corrective action.

## ■ Characteristics of Ca

| | | |
|---|---|---|
| Atomic weight | 40.078 | |
| Melting point | 838 ˚C ($CaCl_2$ 774 ˚C, $Ca(NO_3)_2$ 561 ˚C, $CaSO_4$ 1450 ˚C) | |
| Boiling point | 1480 ˚C ($CaCl_2$ 1600 ˚C) | |
| Oxidation number | +2 $CaO$, $CaO_2$, $Ca(OH)_2$, $CaF_2$, $CaCl_2$, $CaC_2$ | |
| Solubility | $CaCl_2$ | 74.5 g/100 g water (20 ˚C) |
| | $Ca(NO_3)_2$ | 129.9 g/100 g water (20 ˚C) |
| | $CaSO_4$ | 0.298 g/100 g water (20 ˚C) |

Reference:
New Knowledge About Element 111,
Dictionary of Physics and Chemistry, etc.

## ■ Analysis Wavelengths of Ca

| nm | Sensitivity Ratio |
|---|---|
| 422.7 | 1.0 |
| 239.9 | 0.005 |

## ■ Flame Analysis of Ca

Ca, like the other alkaline-earth metals Mg, Sr and Ba, is an element that is easily affected by ionization interference and chemical interference. A typical example of chemical interference is the influence of phosphate in Ca flame analysis, in which Ca and phosphate become bound in the flame to form calcium phosphate, a molecule that does not dissociate easily. Measures that are used to address the problem of interference in flame measurement include the use of (1) standard addition measurement, (2) interference inhibitors, (3) an $N_2O$-$C_2H_2$ flame, etc. Although the standard addition method is widely used to address interference, it may be difficult to obtain accurate analysis results when analyzing samples associated with excessive interference and markedly decreased sensitivity. La is typically used as an interference inhibitor, in which case it is added to both the sample and standard solution at concentrations of several thousand ppm. However, the effectiveness of the inhibitor may not be adequately demonstrated in cases where the obstructing component is present in large quantities. As for use of the $N_2O$-$C_2H_2$ flame, the bonds of a compound that does not easily dissociate are severed at its high temperature of 3000 ˚C, thereby suppressing the interference. However, since the alkaline elements and alkaline-earth elements are easily ionized at high temperature, this can cause reduced sensitivity. When the influence of the coexisting components in the sample matrix is extensive, a combination of the above-mentioned measures may also be effective.

One substance that exhibits interference in the analysis of Ca is Al. Fig. 1 - 6 show the calibration curves generated using various combinations of the above measures in this analysis, including the use of 3 types of standard solutions (0, 0.5, 1 and 2 ppm) with addition of La at 2000 ppm, addition of Al at 10 ppm, and addition of Al and LaA at 10 ppm and 2000 ppm, respectively. Furthermore, each of these was analyzed using an Air- $C_2H_2$ flame and an $N_2O$-$C_2H_2$ flame. It is clear from the results that Al interference is effectively controlled with the addition of La using either type of flame.

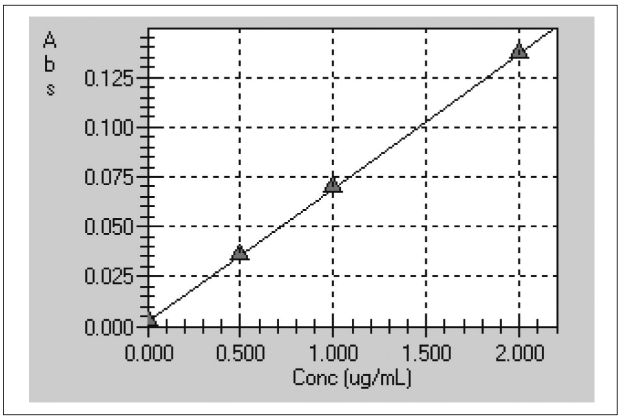


**Fig.1 Calibration Curve (Add La, Air-$C_2H_2$)**

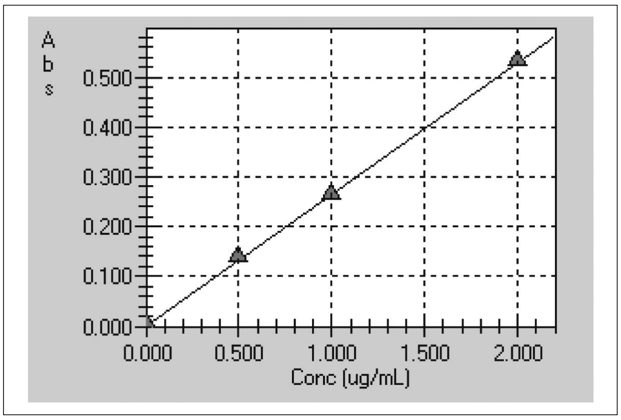


**Fig.2 Calibration Curve (Add Al, Air- $C_2H_2$)**

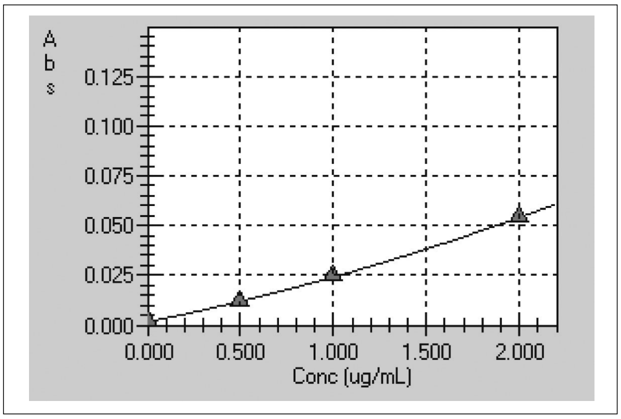


**Fig.3 Calibration Curve (Add Al/La, Air- $C_2H_2$)**

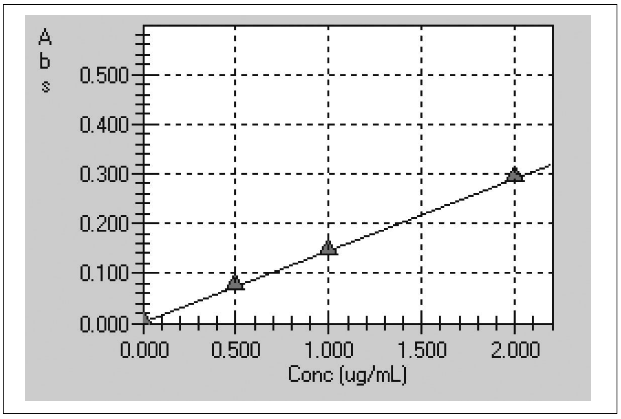


**Fig.4 Calibration Curve (Add La, $N_2O$-$C_2H_2$)**

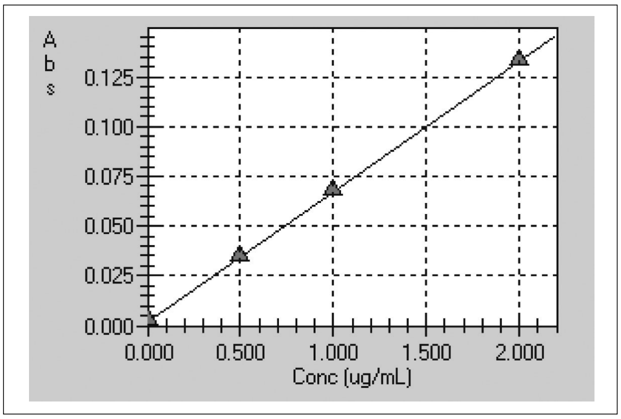


**Fig.5 Calibration Curve (Add Al, $N_2O$-$C_2H_2$)**

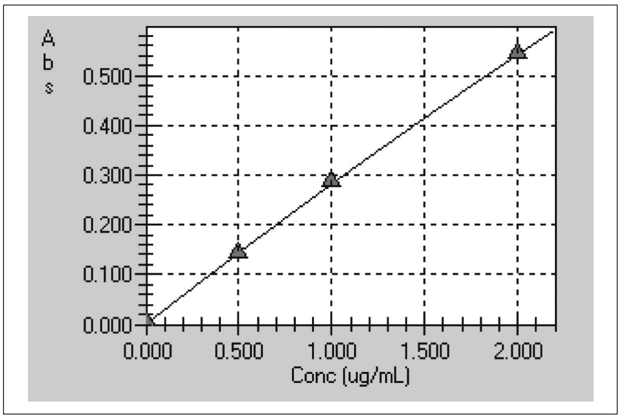


**Fig.6 Calibration Curve (Add Al/La, $N_2O$-$C_2H_2$)**

## ■ Conclusion

Here we introduced the analysis of Ca in talc according to the method prescribed by the Japanese Pharmacopeia, in which the addition of La is used in conjunction with the $N_2O$-$C_2H_2$ flame. Talc (hydrous magnesium silicate) may contain chlorite (hydrous aluminum magnesium silicate), magnesite (magnesium carbonate), calcite (calcium carbonate), and dolomite (calcium magnesium carbonate) as related minerals. An interference inhibitor was used in conjunction with a high-temperature flame to remove the interference due to these coexisting substances in the sample.



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