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Optimize Determination of Calcium Using Flame Atomic Absorption

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Abstract:

In this study the absorbance of 5 ppm calcium solution was determined at 422.67 nm. This absorbance was compared with the absorbance of the solution when treated with phosphate, strontium, sodium chloride solutions. It is found that the absorbance of standard solution of calcium is 0.1201, and for other treatments 0.0840, 0.1101, 0.1339 respectively. The effect of organic solvent (ethanol) was also studied.

Key words: determination of calcium, atomic absorption

INTRODUCTION

Calcium is a chemical element with symbol Ca and atomic number 20. Calcium is a soft gray alkaline earth metal, fifth-most-abundant element by mass in the Earth's crust. The ion Ca^{2+} is also the fifth-most-abundant dissolved ion in seawater by both molarity and mass, after sodium, chloride, magnesium, and sulfate.[1] Free calcium metal is too reactive to occur in nature. Calcium is produced in supernova nucleosynthesis.

Calcium is essential for living organisms, in particular in cellphysiology, where movement of the calcium ion into and out of the cytoplasm functions as a signal for many cellular processes. As a major material used in mineralization of bone, teeth and shells, calcium is the most abundant metal by mass in many animals.

It is a silvery metallic element that must be extracted by electrolysis from a fused salt like calcium chloride.[2] Once produced, it rapidly forms a gray-white coating of calcium oxide and calcium nitride for reacting with the oxygen and nitrogen in the air when exposed to it. In bulk form (typically as chips or "turnings"), the metal is somewhat difficult to ignite, more so even than magnesium chips; but, when lit, the metal burns in air with a brilliant high-intensity orange-red light. Calcium metal reacts with water, generating hydrogen gas at a rate rapid enough to be noticeable, but not fast enough at room temperature to generate much heat, making it useful for generating hydrogen.[3] In powdered form, however, the reaction with water is extremely rapid, as the increased surface area of the powder accelerates the reaction with the water. Part of the reason for the slowness of the calcium–water reaction is a result of the metal being partly protected by insoluble white calcium hydroxide; in water solutions of acids, where this salt is soluble, calcium reacts vigorously.Calcium salts are colorless from any contribution of the calcium, and ionic solutions of calcium (Ca^{2+}) are colorless.[4]. Calcium has two allotropes [5], higher electrical resistivity than copper or aluminium [6], hazardous and sometimes gives violent reaction with water and acids compared with other metals [7]. Calcium ions and most of its compounds have low toxicity due to high natural abundance in the environment and organisms [8-10].

Types of Interferences in Atomic Absorption Spectroscopy

Classification and Causes

An interference is a phenomenon that affects the measurement or the population of ground state atoms of an analyte element.

Spectral Interferences

Spectral interference is caused by radiation overlap of absorption line due to emissions from another element or compound.

If an absorbing wavelength of an element , not being determined but present in the sample falls within the measuring line of the element of interest, the absorbance of the element will be measured together with the analyte of interest and give a higherabsorbance value.

Interference due to overlapping lines is rare in AAS.

Spectral interference is also affected by the radiation of an emission line of another element or compound, or background radiation from flame, solvent or sample.

Chemical Interferences

Chemical interference occurs when an analyte is not totally decomposed in flame. There is less atoms present, and therefore a reduced absorbance of the analyte. Anions SO_4 and PO_4 combines with metal to form stable compounds and decrease the absorption of the analyte.

Calcium signal is depressed due to formation of $CaSO_4$ or $CaPO_4$.

Ionization Interferences

Ionization interference affects group oneand twoonly. These include Ba, Ca, Sr, Na, K.

This occurs only in hot flames. The energy of the flame excite the ground state atoms to ionic state by loss of electron resulting in a depletion of ground state atoms, and the element does not absorb at the correct wavelength.

Matrix Interferences

Matrix interference is a physical interference, and can either suppress or enhance absorbance signal of analyte. It occurs when components of sample matrix other than the analyte react to form molecular species and sample background.

The detector picks up unspecified signals from sample matrix that do not match the absorbance line of the analyte. This results in spurious readings that can affect quantitative and qualitative analysis.

Causes:

1. Characteristics of sample and standards differ in viscosity and surface tension.
2. Sample and standards are prepared in different solvents.
3. Sample and standards are measured at different temperatures.
4. Sample contains a high concentration of dissolved salts or acid.
5. Organics are present in sample matrix.
6. Sample and standards differ in aspiration and atomization rate in flame.[5]

EXPERIMENTAL AND METHODS

- A solution containing 5ppm Ca was prepared and the absorbance at 422.67 nm was measured.

-A solution containing 5ppm Caand 10 ppm phosphate was prepared and the absorbance was measured at 422.67 nm.

- A solution containing 5ppm Ca,10 ppm phosphate ,and 1% $SrCl_2$ was prepared and the absorbance was measured at 422.67 nm.

- A solution containing 5ppm Caand 1000 ppm NaClwas prepared and the absorbance was measured at 422.67 nm and compared with only 5ppm Ca.

- A solution containing 5ppm Ca in 50 % ethanol was prepared and the absorbance was measured at 422.67 nm, then compared with 5ppm Ca in water. The effect of organic solvent was studied.

RESULTS

Table (1): Determination of absorbance of calcium solution

	Sample solutions	**Absorbance at 422.67 nm**
1	5 ppm Ca	0.1201
2	5 ppm Ca +10 ppm Phosphate	0.0840
3	5 ppm Ca + 10 ppm Phosphate + 1% Sr Cl_2	0.1101
4	5 ppm Ca +1000 ppm NaCl	0.1339
5	5 ppm Ca +50% Ethanol	0.1412

DISCUSSION

Chemical interference: means reaction between the element to be analyzed with another elements or compounds. This affects atomization process, so affects the absorption and emission. An example of this type of interference formation of refractory compounds, for example determination of calcium in presence of phosphate ions, calcium phosphate is formed which is hardly atomize .This shown in table (1) in which the absorbance of 5 ppm Ca is 0.1201 at 422.67 nm , this absorbance is decreased when treated with sodium phosphate solution to 0.0840 .This type of interferences can be eliminated by using releasing agent which is cation like Lanthanum or strontium to decrease the effect of phosphate ions by forming stable compound.

$$Ca_3(PO_4)_2 + 6Sr \longrightarrow 2Sr_3(PO_4) + 3Ca$$

This will increase the concentration of calcium in solution. From table (1), absorbance of 5 ppm Ca + 10 ppm phosphate is 0.0840, after addition of SrCl2 solution the absorbance increases to 0.1101.

Physical interference: most parameters that affect the rate of sample uptake in burner and atomization efficiency can be considered physical interferences, this include variation in sample viscosity, changes in flame temperature and solvent variation. From table (1), absorbance of 5 ppm Ca/H_2O is 0.1201, this increases to 0.1412 when 50% ethanol is used as solvent.

Ionization interferences: an appreciable fraction of alkali, alkaline earth element may be ionized in the flame. Since we are measuring the unionized atoms, either emission or absorption signals will be decreased. However the presence of other easily ionized elements in the sample will add free electrons to the flame and suppress ionization of the test element.

$$\mathrm{Ca} \rightleftharpoons \mathrm{Ca}^{2+} + 2\bar{e}$$

$$n\mathrm{Na} \longrightarrow n\mathrm{Na}^{+} + n\bar{e}$$

$$\mathrm{Ca}^{2+} + n\bar{e} \longrightarrow \mathrm{Ca}$$

In the table of the result absorbance of 5 ppm Ca is 0.1201 was increased by adding NaCl solution to give absorbance A 0.1339.

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