IS 5949 (1990): Methods for volumetric determination of calcium and magnesium using EDTA [CHD 1: Inorganic Chemicals]

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IS 5949 : 1990
(Reaffirmed 2003)

भारतीय मानक

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(दूसरा पुनरीक्षण)

Indian Standard

METHODS FOR VOLUMETRIC DETERMINATION OF CALCIUM AND MAGNESIUM USING EDTA

(Second Revision)

543·24 : 546·41 + 546·46

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 1

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 31 May 1990, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1970 and revised in 1980. During the first revision, the Patton and Reeder's indicator method for determination of calcium had been added. In the present revision GbHA indicator method for determination of calcium has been deleted and the test methods have been modified in the light of ASTM D 1126-1986 'Standard test method for hardness in water' issued by the American Society for Testing and Materials.

EDTA method for the determination of calcium and magnesium is being used extensively in the analysis of commercial materials, such as cement, leather, paints, petroleum products, slags, ores and rocks, water, pharmaceuticals, food and biological products.

The methods prescribed in this standard are intended to be of general nature. The method of preparing the solution of the material is not covered in this standard. It has to be done as prescribed in the individual material specification. The test solution taken for determination is generally expected to be free from interfering ion(s) or radical(s). However, interference of small amounts of iron, aluminium, copper — either alone or combined — which is normally associated with calcium and magnesium, may be overcome by using masking agents like potassium cyanide, triethanolamine and hydroxylamine hydrochloride either alone or in combination. Alkali cyanides are found to be useful for masking common interfering ions like zinc, cadmium, copper, mercury, nickel, cobalt, silver and platinum at *p*H above 8 when determinations of calcium and magnesium are carried out individually.

The determination of calcium by precipitating it as oxalate and titrating with standard potassium permanganate is covered in IS 4285 : 1967 'Method for volumetric determination of calcium'.

This standard intends to achieve uniformity of the method of volumetric determination of calcium and magnesium prescribed in Indian Standards for analysis of chemical products.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

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Indian Standard

METHODS FOR VOLUMETRIC DETERMINATION OF CALCIUM AND MAGNESIUM USING EDTA

(Second Revision)

1 SCOPE

1.1 This standard prescribes the methods for the volumetric determination of calcium and magnesium using ethylenediamine tetraacetic acid (EDTA).

NOTE — These methods do not cover analysis of metals where limits for various interfering elements and/or radicals are prescribed.

2 REFERENCES

2.1 The Indian Standards listed below are the necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1977	Specification for water for general laboratory use (*second revision*)
4285 : 1967	Method for volumetric determination of calcium

3 QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

4 REAGENTS

4.1 Patton and Reeder's Indicator (P and R Indicator) — 1 Percent (m/v)

Mix 1·0 g of solid indicator [2 hydroxy - 1 - (2 hydroxy - 4 - sulpho - 1 - napthylazo) - 3 - napathoic acid - (HHSNNA)] with 99 g of pure sodium chloride or sodium sulphate anhydrous in a mortar and pestle and grind well to about 250 to 150 micron, mixing it intimately so as to make the diluted reagent powder homogeneous. Preserve in a tightly stoppered amber-coloured bottle.

4.2 Eriochrome Black T Indicator (EBT Indicator)

Dissolve 0·5 g of EBT indicator and approximately 4·5 g of hydroxylamine hydrochloride in 100 ml of rectified spirit (ethyl alcohol or methyl alcohol).

4.3 Standard Zinc Solution, 0·01 M

Dissolve 0·653 8 g of pure zinc dust or granules of 99·9 percent purity in 20 ml of approximately 1 : 1 hydrochloric acid, warm if necessary. Cool and make up to 1 litre exactly in a volumetric flask. Preserve this solution in a tightly closed glass bottle.

4.4 Buffer Solution

Dissolve 70 g of ammonium chloride and 570 ml of 30 percent ammonia solution (relative density 0·88 — 0·90) in water and make up to 1 litre.

4.5 Standard Ethylene Diamine Tetraacetic Acid (EDTA) Solution — (0·01 M)

Dissolve 3·75 g of disodium ethylene diamine tetraacetate dihydrate in water and make up to 1 litre in a volumetric flask. Standardize this with standard zinc solution. Pipette out 25 ml of standard zinc solution in a 250 ml conical flask. Adjust the *p*H to approximately 10 with buffer solution. Dilute to about 100 ml and add 3 to 4 drops of EBT indicator solution. This will give a red colour. Titrate with 0·01 M EDTA solution to a clear blue end point free from violet tinge. This solution will be slightly stronger than 0·01 M. Dilute the solution to exactly 0·01 M by adding calculated amount of water and recheck the strength by titrating 25 ml of standard zinc solution by exactly the same manner as given above. This should consume exactly 25·0 ml of standard EDTA solution.

4.6 Sodium Hydroxide Solution, 20 percent (m/v)

NOTE — Preserve in a polyethylene bottle.

4.7 Triethanolamine Solution, 10 percent (m/v)

4.8 Potassium Cyanide Solution, 10 percent (m/v)

NOTE — Preserve in a polyethylene bottle.

4.9 Hydroxylamine Hydrochloride Solution, 10 percent (m/v)

4.10 Magnesium EDTA Complex Solution

Dissolve 2·465 g of pure magnesium sulphate ($MgSO_4.7H_2O$) in 300 to 400 ml of water. Add to this 3·72 g of sodium EDTA solid. Dissolve and adjust *p*H to 9·5 to 10. Add 8 to 10 drops of EBT indicator solution. The colour should be pink. Titrate this with 0·01 M EDTA solution till the colour changes to pure blue free from violet tinge. Dilute this to exactly 1 litre in a volumetric flask.

5 DETERMINATION OF CALCIUM

5.1 Take a suitable aliquot of the sample solution prepared as prescribed in the material specification and expected to contain approximately 5 to 20 mg of calcium as CaO, in a 500 ml conical flask, Add 25 ml of triethanolamine solution, 10 ml of hydroxylamine hydrochloride solution and 2 ml of potassium cyanide solution. Dilute to about 150 to 200 ml with water and adjust the *p*H to 12·5 to 13 with 20 percent sodium hydroxide solution (4·6) using a *p*H indicator paper or preferably a *p*H meter. Add approximately 0·1 g of Patton and Reeder's indicator mixture and stir to dissolve. Titrate with 0·01 M EDTA solution from a burette, till the colour changes from red to pure blue free from any violet tinge. Towards the end point, the colour turns a voilet which on further dropwise addition gives the pure blue end point. It is easier to note the end point by comparing the colour with that of a previously titrated solution having a pure blue end point, where a slight excess of EDTA has been added.

NOTE — Where the solution containing calcium and magnesium contains ammonium salts, that is, in minerals analysis, the amount of sodium hydroxide solution required to bring the *p*H to 12·5 to 13 will be more. It is important that *p*H must be brought to 12·5 to 13 as otherwise all magnesium will not precipitate, leading to higher titration reading.

5.2 Calculation

$$\text{Calcium (as Ca) percent by mass} = \frac{V_1 \times 0{\cdot}040\,08}{M}$$

where

V_1 = volume in ml of EDTA solution consumed in titration; and

M = mass in g of the sample in the solution taken for the test.

6 DETERMINATION OF MAGNESIUM

6.1 Take a suitable aliquot of the solution of the material exactly the same as under **5.1**, expected to contain approximately 10 to 30 mg of calcium and magnesium combined oxides in a 500 ml conical flask. Add 10 ml of hydroxylamine hydrochloride solution, 2 ml of potassium cyanide solution and 25 ml of triethanolamine solution. Dilute to 150 to 200 ml and add sufficient quantity of buffer solution to bring the *p*H to 9·5 to 10. Add 3 to 4 drops of EBT indicator solution and titrate with 0·01 M EDTA solution till the red colour changes to pure blue end point free from violet tinge. It is easier to note the end point by comparing the colour with that of previously titrated solution having a pure blue end point, where a slight excess of EDTA solution has been added.

NOTE — In case magnesium is not present, add 5 ml of magnesium EDTA complex solution in order to get proper end point. This should be added before titration.

6.2 Calculation

$$\text{Magnesium (as Mg), percent by mass} = \frac{0{\cdot}024\,3 \times (V_2 - V_1)}{M}$$

where

V_2 = volume in ml of EDTA solution consumed in titration;

V_1 = volume in ml of EDTA consumed in titration for calcium determination, in the same aliquot of solution of sample; and

M = mass in g of the sample taken for the test.

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